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EVALUATION OF
ASPHALT-AGGREGATE DISTRIBUTION
IN BITUMINOUS MIXES
BY AUTORADIOGRAPHY

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NO. 1

Joint
Highway
Research
Project

by
D.R. LAMB
W.H. GOETZ
and
J.E. CHRISTIAN

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Technical Paper

EVALUATION OF ASPHALT-AGGREGATE DISTRIBUTION
IN BITUMINOUS MIXES BY AUTORADIOGRAPHY

TO: K. B. Woods, Director
Joint Highway Research Project

December 10, 1962

FROM: H. L. Michael, Associate Director
Joint Highway Research Project

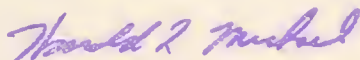
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Project: C-36-21B

Attached is a technical paper entitled "Evaluation of Asphalt-Aggregate Distribution in Bituminous Mixes by Autoradiography" which has been authored by D. R. Lamb, W. H. Goetz, and J. E. Christian. The paper will be presented at the Annual Meeting of The Association of Asphalt Paving Technologists in San Francisco, February 13-20, 1963.

The paper is a summary of the research performed at Purdue University by Dr. Lamb under the guidance of Professors Goetz and Christian. The object of the paper is to illustrate how radioactive isotope techniques can be applied to the investigation of some of the fundamentals of the phase relationships in bituminous mixes.

It is requested that approval of publication of the paper by the AAPT be given.

Respectfully submitted,



Harold L. Michael, Secretary

HLH/lkc

Attachments

Copy:	F. L. Ashbaucher	F. S. Hill	R. E. Mills
	J. R. Cooper	G. A. Leonards	H. B. Scott
	W. L. Dolch	J. F. McLaughlin	J. V. Smythe
	W. H. Goetz	R. D. Miles	J. L. Waling
	F. F. Havey		E. J. Yoder

Technical Paper

EVALUATION OF ASPHALT-AGGREGATE DISTRIBUTION IN BITUMINOUS
MIXES BY AUTORADIOGRAPHY

by

Donald R. Lamb

William H. Goetz

and

John E. Christian

Joint Highway Research Project

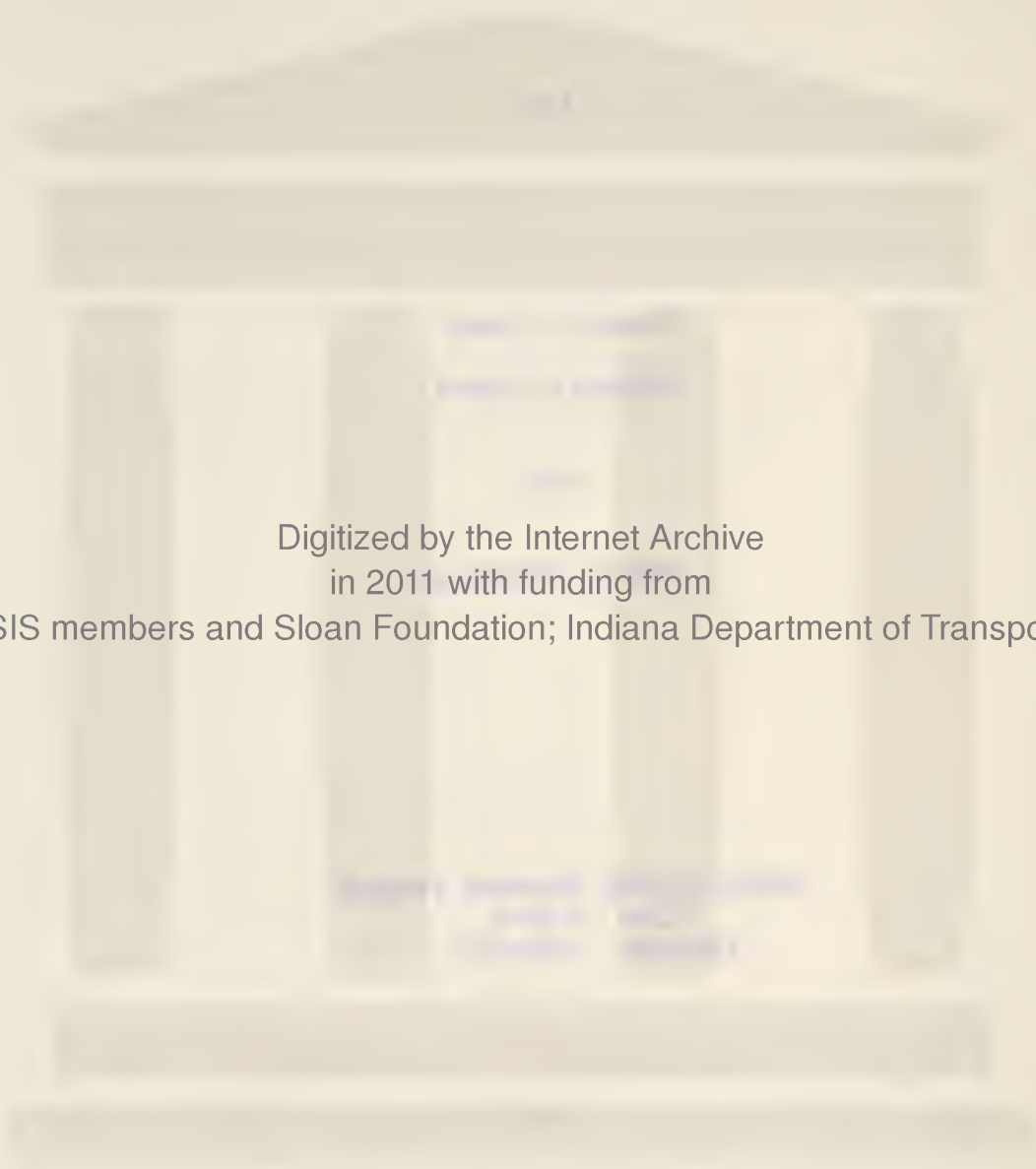
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Purdue University
Lafayette, Indiana

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THE UNIVERSITY OF CHICAGO
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DEPARTMENT OF CHEMISTRY



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EVALUATION OF ASPHALT-AGGREGATE DISTRIBUTION IN BITUMINOUS MIXES BY AUTORADIOGRAPHY

INTRODUCTION

Radioactive isotopes have not been used for basic research in the area of bituminous mixtures to any great extent. The determination of asphalt content in a bituminous mixture (1)¹, the use of an isotope to determine the amount of stripping of asphalt from aggregate (2), the measurement of mixing efficiency using a radioactive isotope (3) and some unpublished reports on density and asphalt content are the only bituminous research areas in which isotopes have been used. It is the object of this paper to illustrate how radioactive isotope techniques can be applied to the investigation of some of the fundamentals of the phase relationships in bituminous mixes.

In the interest of brevity the authors have not reviewed the fundamentals of radioactivity and autoradiography. The reader is referred to basic texts in this area.

MATERIALS AND PROCEDURES

The primary aggregate used in this research was crushed limestone. In some phases of the work, a river gravel and Belgium black marble were used. A naturally radioactive aggregate, Wyoming urananite, which gave off alpha, beta, and gamma emissions, was used also. The asphalt used was a 60-70 penetration grade asphalt cement.

¹ Numbers in parentheses refer to selected references.

The radioisotope chosen for the research was sulfur-35. The properties of the isotope that were compatible with the research are as follows: low beta emission energy giving good contrast in autoradiography; a relatively short half-life making reuse of all equipment possible; and, as elemental sulfur in benzene, easy dispersability in asphalt.

After receiving the sulfur-35 in benzene, a precalculated amount of the nonradioactive isotope of sulfur in benzene was added to the purchased mixture as a carrier for the radioactive sulfur. This was done so that a uniform distribution of the sulfur would be made in the asphalt. The amount of benzene and carrier that were added was calculated so that 500 λ of final mixture of benzene, carrier and sulfur-35 would have one μC of activity as of the day of preparation. The 500 λ of mixture was then added to 50 grams of the asphalt cement at 275°F and mixed by hand with a tongue depressor until it was too viscous to mix any longer. The treated asphalt was reheated to 275°F, mixed slightly again, and then placed in the mixing bowl with the aggregate.

Hand mixing was used for all parts of the study except when mixing time and viscosity of the asphalt were variables. In these cases, machine mixing with a modified Hobart Mixer was used. Specimens were formed using 50-blow Marshall compaction.

A considerable amount of effort was spent in this study in achieving good autoradiographic definition. This was the basic measuring device for this research. The method of sample preparation that was finally utilized was to take the specimen from the mold and place it in the freezing compartment of a regular refrigerator. After 24 hours it was cut with a diamond saw and then polished with a 100, 120, 240, and 320 grit aluminum oxide paper. The 100 and 320 grit papers were used in hand polishing and the 120 and 240 grit

papers were used on a belt sander. This gave a smooth plane surface that could be brought into full contact with the photographic emulsion during the exposure phase of the work.

Many different photographic emulsions were investigated also and the two that were finally found acceptable were: EK Medical X-ray film, Blue Brand and Kodak Autoradiographic Stripping Film Type NTB (emulsion number FW-1826-1). The X-ray film was used generally in the research, but where increased definition was desired the NTB film was used. Another reason for doing the bulk of the autoradiographic work on the X-ray film was the relative costs of the two emulsions. The NTB emulsion was 45 times as expensive as the X-ray film.

RESULTS

The great amount of work that went into the initial phase of the investigation to produce good autoradiographic techniques is omitted here in the interest of brevity. A full description can be found in reference (4). However, the degree of definition that could be obtained in the autoradiographs was of prime importance. Therefore, in order to investigate the definition obtained in more detail than could be done with them in the natural size, an overhead projector was used to project the autoradiograph image on a screen. An increase of 20 diameters was very convenient for the room and screen size. However, photomicrographs were made for presentation in manuscript form.

A digression is in order at this point to explain how the autoradiographs and the other photographs are handled in this presentation. Due to the fact that the asphalt, generally speaking, was radioactive, the film actually showed a positive picture. Thus, when viewing the film the darkened area represents the asphalt and the light area the limestone aggregate.

In contrast to the autoradiograph, the film in a reflected light picture is a negative. To keep as much definition as possible in all pictures a contact print was made in all cases whether autoradiograph or reflected light photography was used. This made the prints of the autoradiographs actually negatives and those of the regular photographs positives. The reader must school himself to this difference if the figures in this presentation are to be meaningful.

A recognizable position was chosen on the polished surface of a specimen and a photomicrograph was made of this area. Then the autoradiograph was placed under the microscope and the same area was again photographed. Care was taken so that the same area was covered in each instance. The two photomicrographs could then be compared as to definition. Figure 1 shows an autoradiograph for an asphalt-aggregate specimen using a graded limestone showing the area chosen for the photomicrograph. The small circle outlines this area. Figure 2 is the photomicrograph of the face of the sample, and Figure 3 is the photomicrograph of the autoradiograph. Due to the fact that both photomicrographs were taken from positives, negatives were made and positive prints for the two figures were produced.

In comparing the two photomicrographs it can be seen that there is more detail shown from the print of the autoradiograph than from the one of the specimen. Also notice that the emulsion grains show in the photograph from the autoradiograph because the picture was made from a film.

Compaction in the Marshall apparatus produced some degradation of the coarse aggregate. This degradation occurred when the coarse material was not cushioned by a matrix. The hot bituminous material was then forced into many of the fractures caused by the rupturing of the coarse aggregate. When the autoradiograph was made from a section through one of these asphalt-filled fractures the result was recorded by the film.

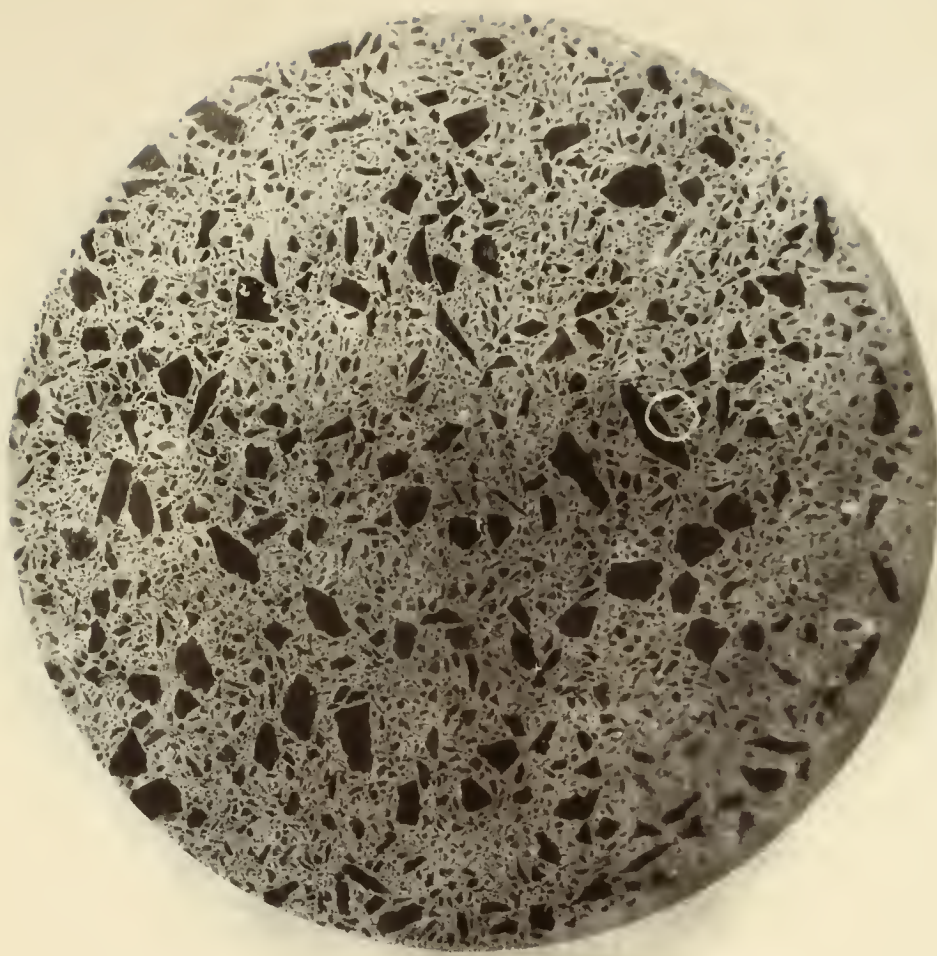


Figure 1 Autoradiograph Showing Position of
Photomicrographs (Example 1.)



Figure 2 Photomicrograph of Specimen Face — Position
Shown in Figure 1 (x 20)

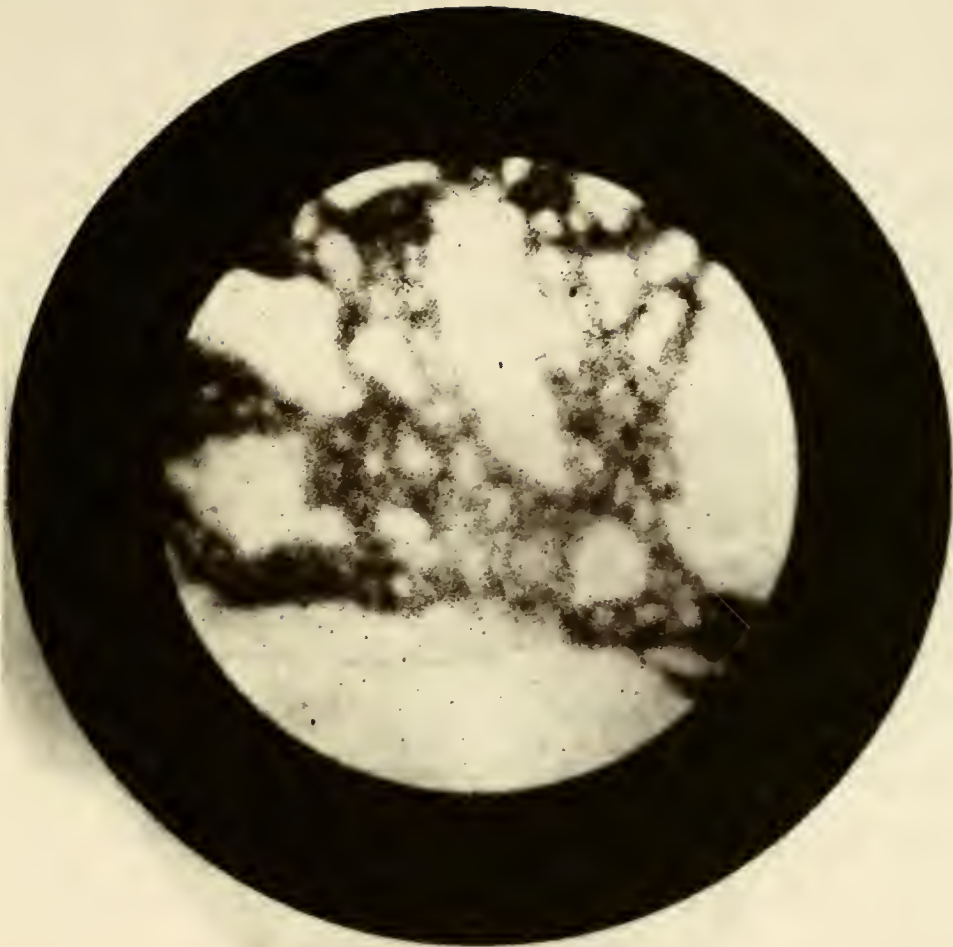


Figure 3 Photomicrograph of Autoradiograph — Position
Shown in Figure 1 (x 20)

A regular photograph of the sample face also revealed the impregnated fissure when light-colored aggregate was used. However, when a dark aggregate was used, Belgium black marble, only the autoradiograph gave the true picture of the asphalt distribution. Figure 4 is the autoradiograph from a graded limestone-aggregate specimen with Figure 5 the companion photograph of the same specimen. Figure 6 is the autoradiograph from the Belgium black marble aggregate specimen and Figure 7, is the photograph of the same specimen surface.

It is also important to note that the photographs show how the stone was broken and scarred by the diamond saw while the autoradiographs do not show these imperfections.

To determine the influence of aggregate gradation on asphalt-aggregate distribution, a fixed gradation of coarse aggregate and a fixed gradation of fine aggregate were blended in various proportions. The coarse fraction chosen had 80 percent of this fraction between the $3/4$ and $1/2$ inch sieves and 20 percent between the $1/2$ and $3/8$ inch sieves. A series of mixes was then made to see how much asphalt would adhere to the coarse material. Mixing temperature for mixes in this part of the research was 275°F .

Mixes were made with from 1 to 6 percent asphalt by weight of the coarse aggregate. A Marshall sample was made and the mixing bowl was weighed back to see how much asphalt was actually on the aggregate. This procedure determined that the coarse aggregate retained about 4 percent asphalt by weight of aggregate.

The gradation chosen for the fine aggregate is shown in Table 1. The amount of asphalt that this gradation would carry was found by making Marshall specimens using 5 percent asphalt by weight of the aggregate and increasing percentages by increments of 1 percent until 9 percent was reached. The specimens were then evaluated by the normal Marshall test and the optimum .

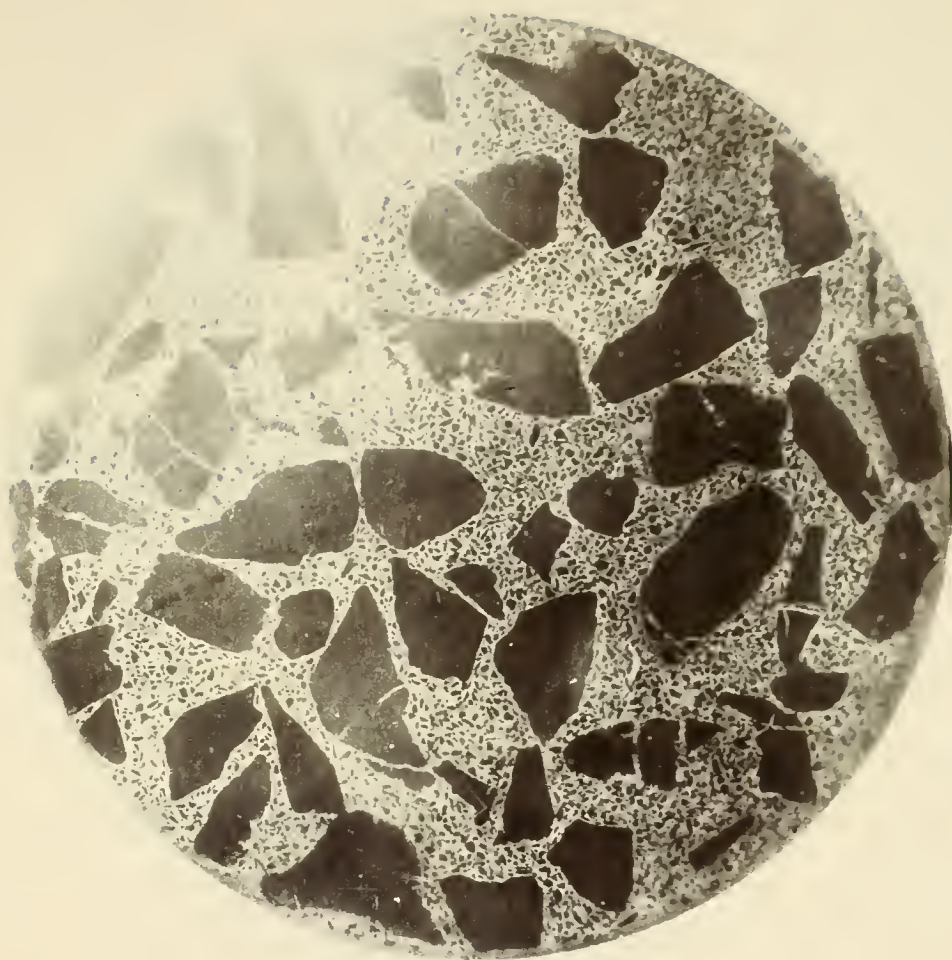


Figure 4 Autoradiograph of a Limestone-Aggregate Specimen

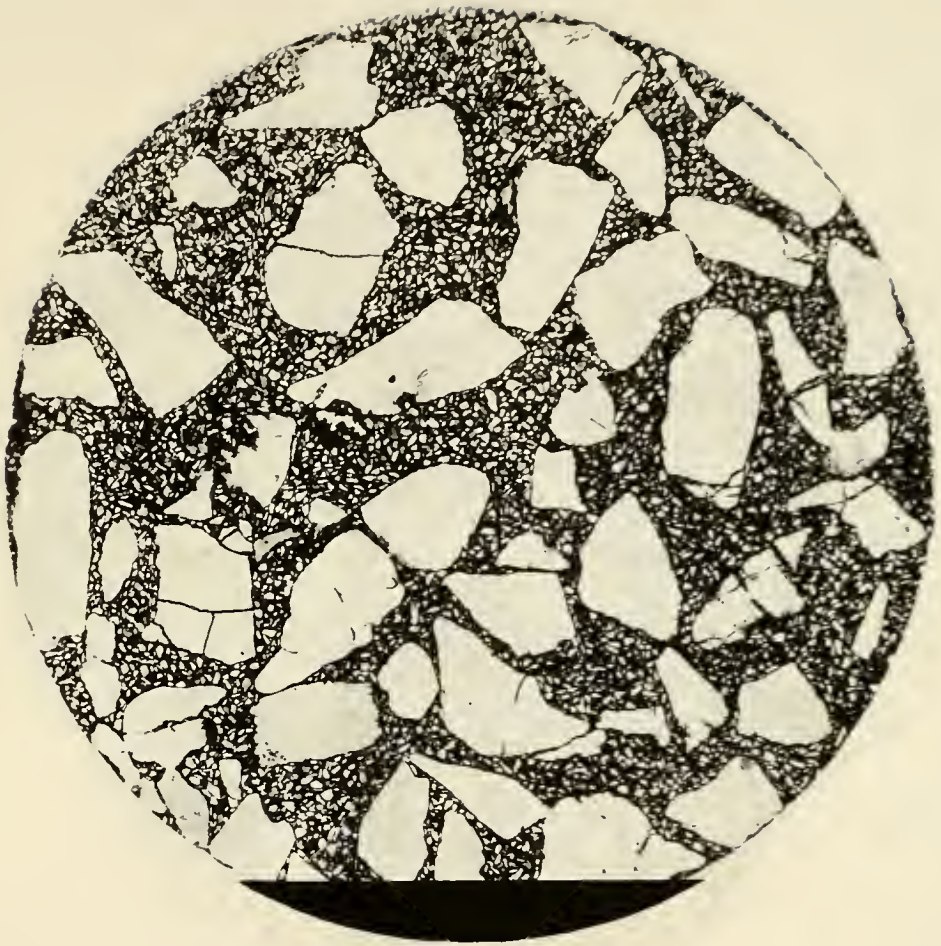


Figure 5 Photograph of Specimen Shown in Figure 4



Figure 6 Autoradiograph of a Belgium-Black-Aggregate Specimen



Figure 7 Photograph of Specimen Shown in Figure 6

percentage chosen from this procedure. The asphalt content chosen for the fine aggregate was 6 percent.

Eleven mixes were then made beginning with 100 percent coarse aggregate and zero percent fine aggregate. This mix composition was changed by increments of 10 percent until the mix contained zero percent coarse aggregate and 100 percent fine aggregate. The asphalt content was determined on the basis of the percent of coarse and fine aggregate used. In the 50-50 mix the total asphalt content was 50 percent of the 4 percent asphalt content of the coarse aggregate plus 50 percent of the 6 percent asphalt content of the fine aggregate, or 5 percent.

The autoradiographs of the complete series beginning with the coarse aggregate only and ending with the fine aggregate only are presented in Figures 8 through 18. In the coarse part of this series the area that is crosshatched is the cement slurry that was used to bind the specimens together for the cutting and polishing process. Only the specimens from 100 percent coarse aggregate through 70 percent coarse aggregate needed to be saturated with the cement slurry in order to be cut. All the rest of the mixes formed a stable specimen.

One phase of this investigation used radioactive Wyoming urananite aggregate to study asphalt-aggregate distribution. When the Wyoming urananite was used as the entire aggregate there was so much activity that the autoradiographs were very low in definition. The amount of Wyoming urananite incorporated into the specimen was then changed so that only one size fraction at a time was used to see how this size was distributed throughout the specimen. The first step was to make a specimen using only untagged asphalt and filler (Wyoming urananite). The amounts of asphalt and filler used were the same throughout all of the following mixes. A two-inch specimen was made

TABLE 1

Gradation of Fine Aggregate

Sieve Sizes	Percent Between
<hr/>	
No. 6 to No. 8	5
No. 8 to No. 16	30
No. 16 to No. 30	26
No. 30 to No. 50	26
No. 50 to No. 100	10
No. 100 to No. 200	2
Passing No. 200	1

100

100

100

100

100

100

100

100

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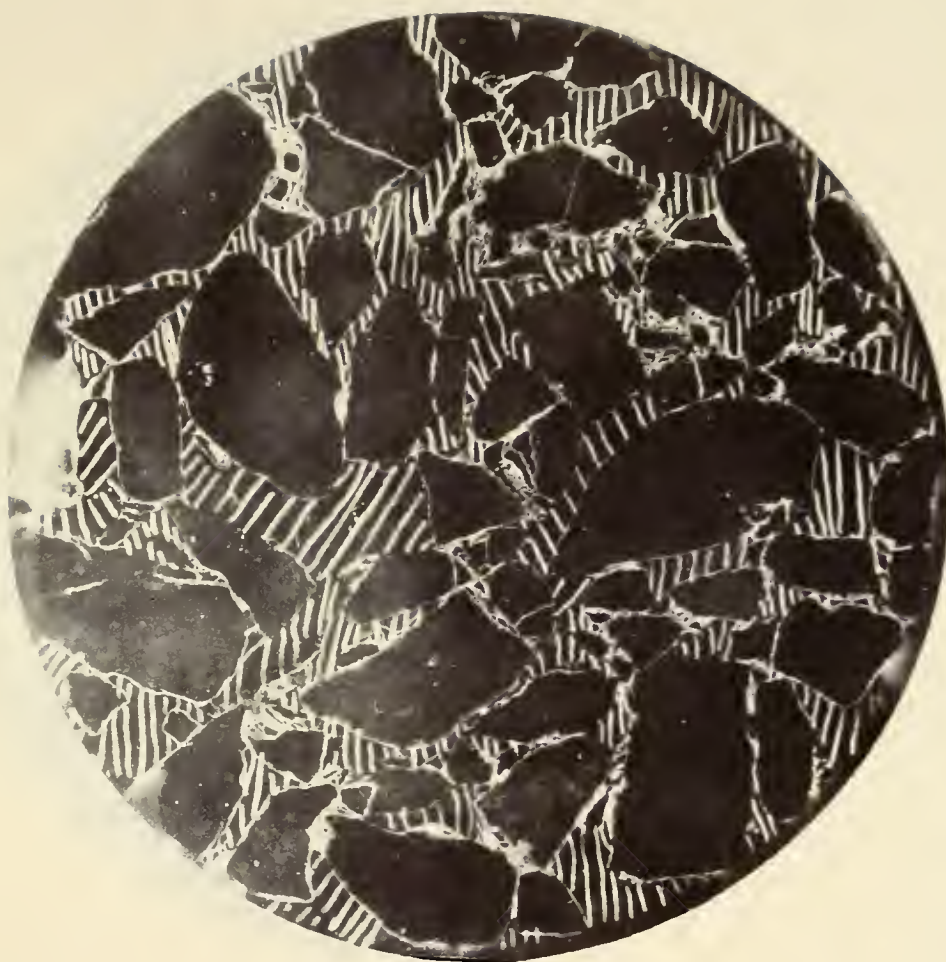


Figure 8 Autoradiograph of Specimen Containing All
Coarse Aggregate

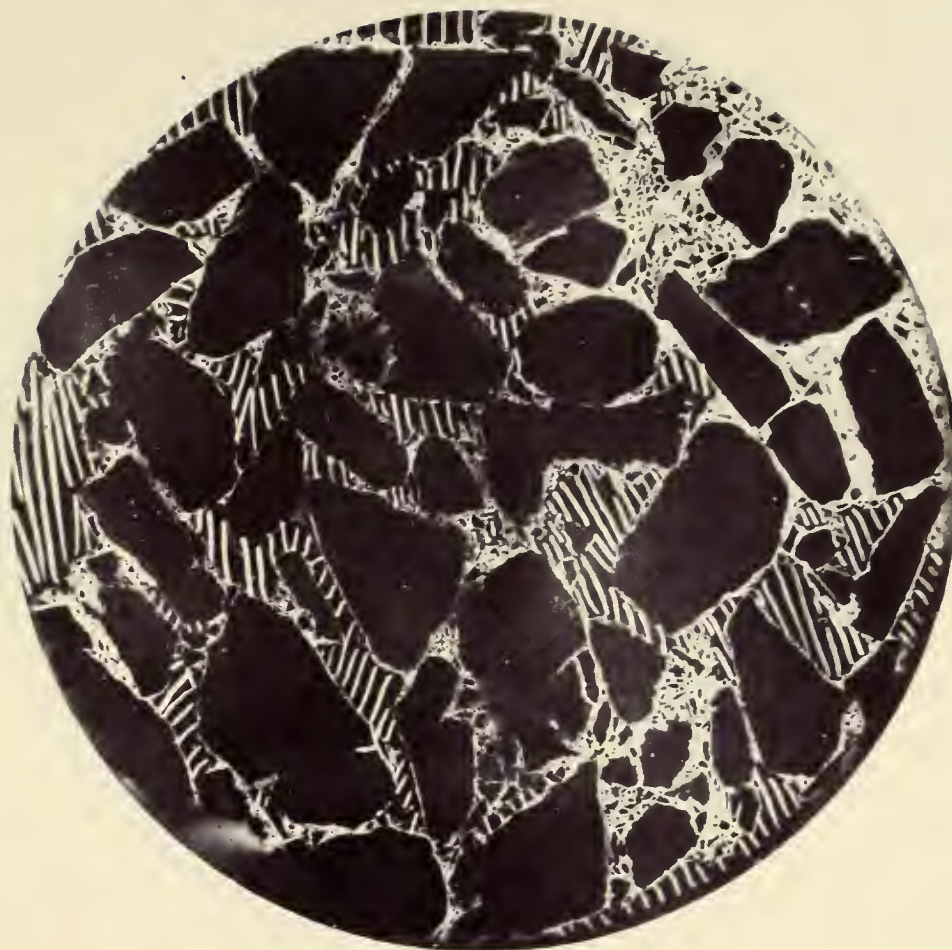


Figure 9 Autoradiograph of Specimen With 90 Percent
Coarse Aggregate and 10 Percent Fine Aggregate

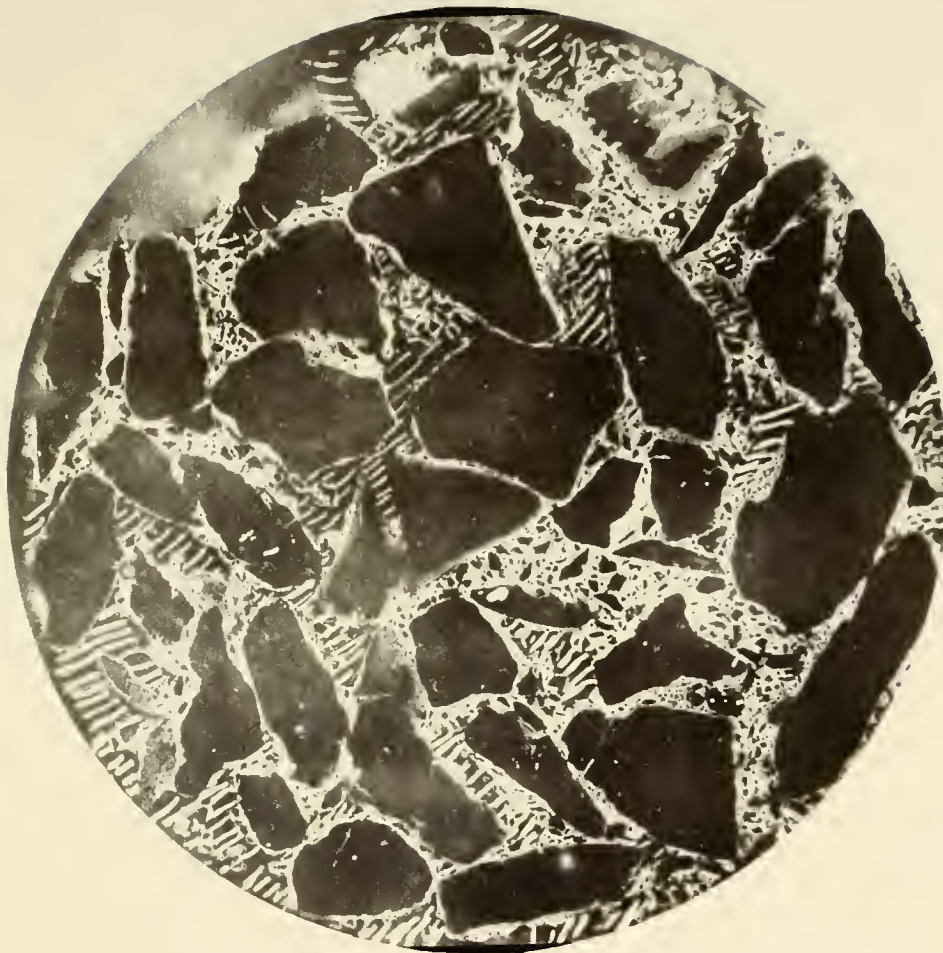


Figure 10 Autoradiograph of Specimen With 80 Percent
Coarse Aggregate and 20 Percent Fine Aggregate

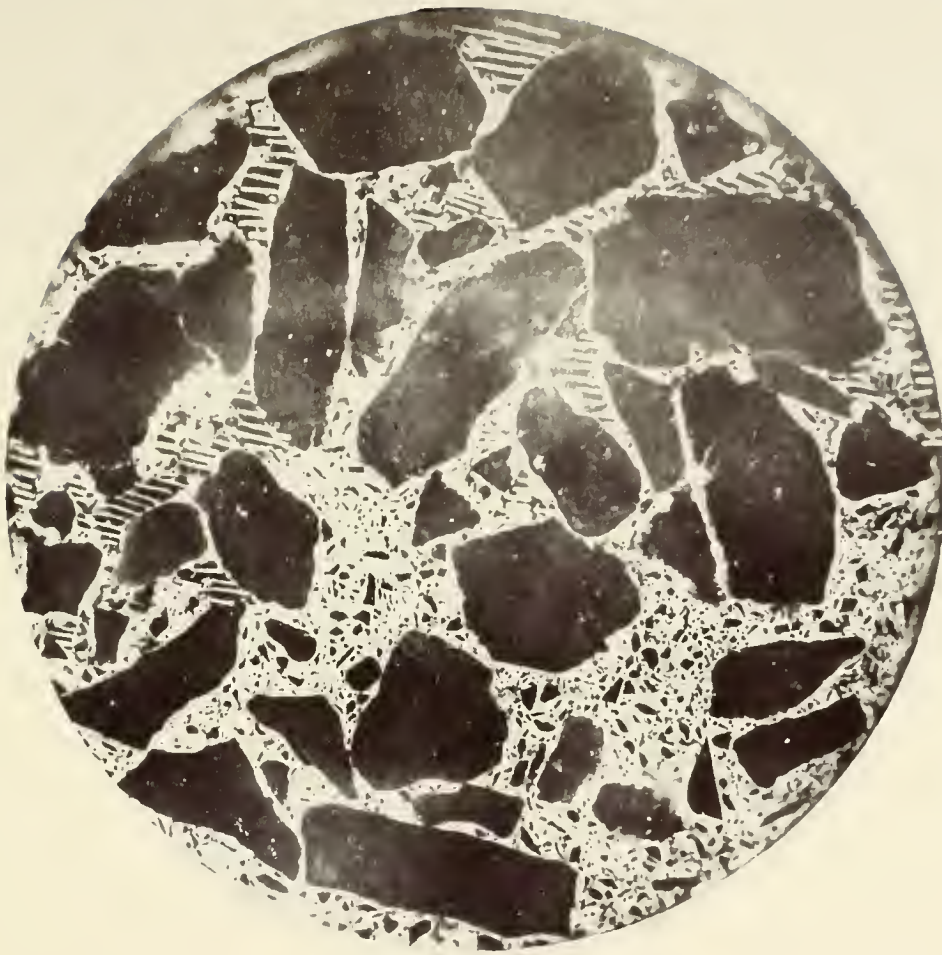


Figure 11 Autoradiograph of Specimen With 70 Percent
Coarse Aggregate and 30 Percent Fine Aggregate

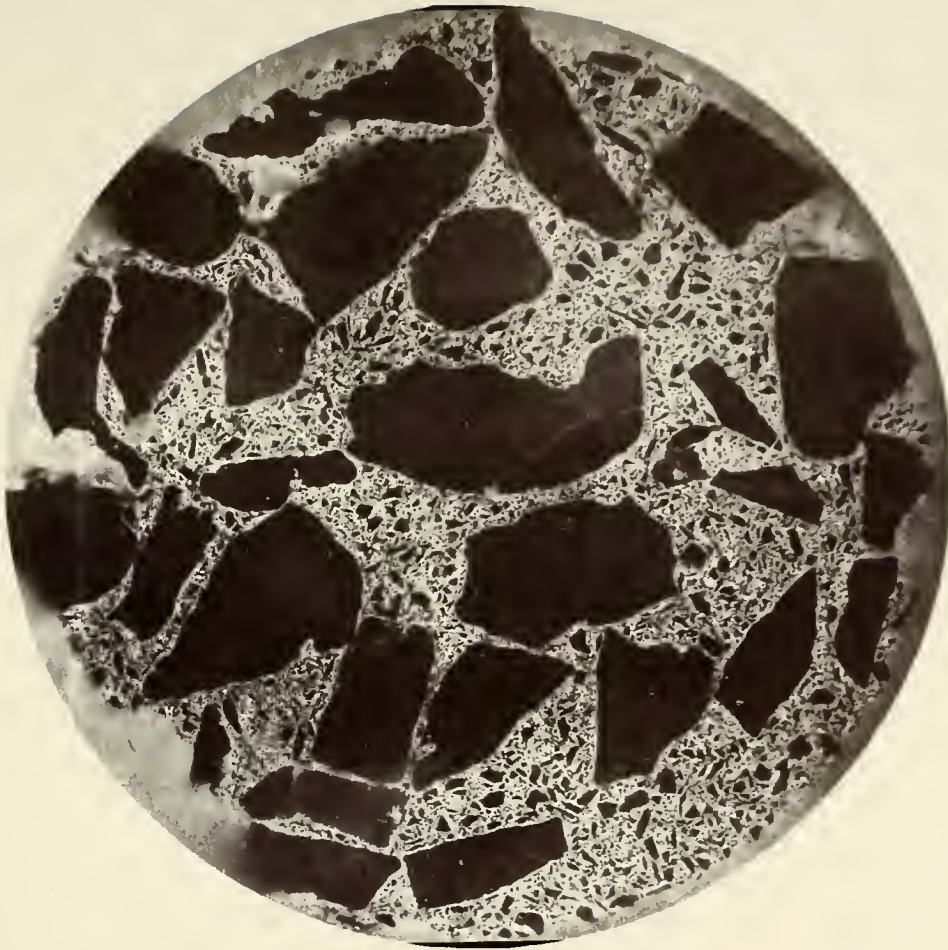


Figure 12 Autoradiograph of Specimen With 60 Percent
Coarse Aggregate and 40 Percent Fine Aggregate

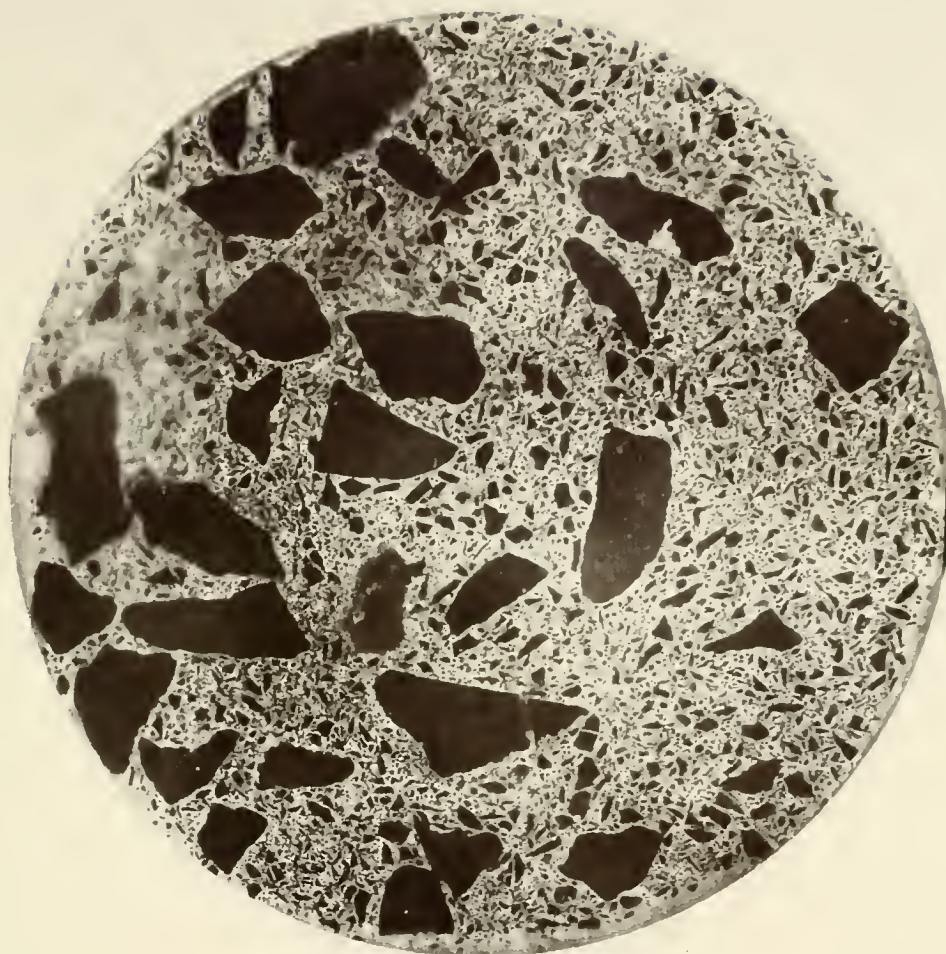


Figure 13 Autoradiograph of Specimen With 50 Percent of
Both Coarse and Fine Aggregate

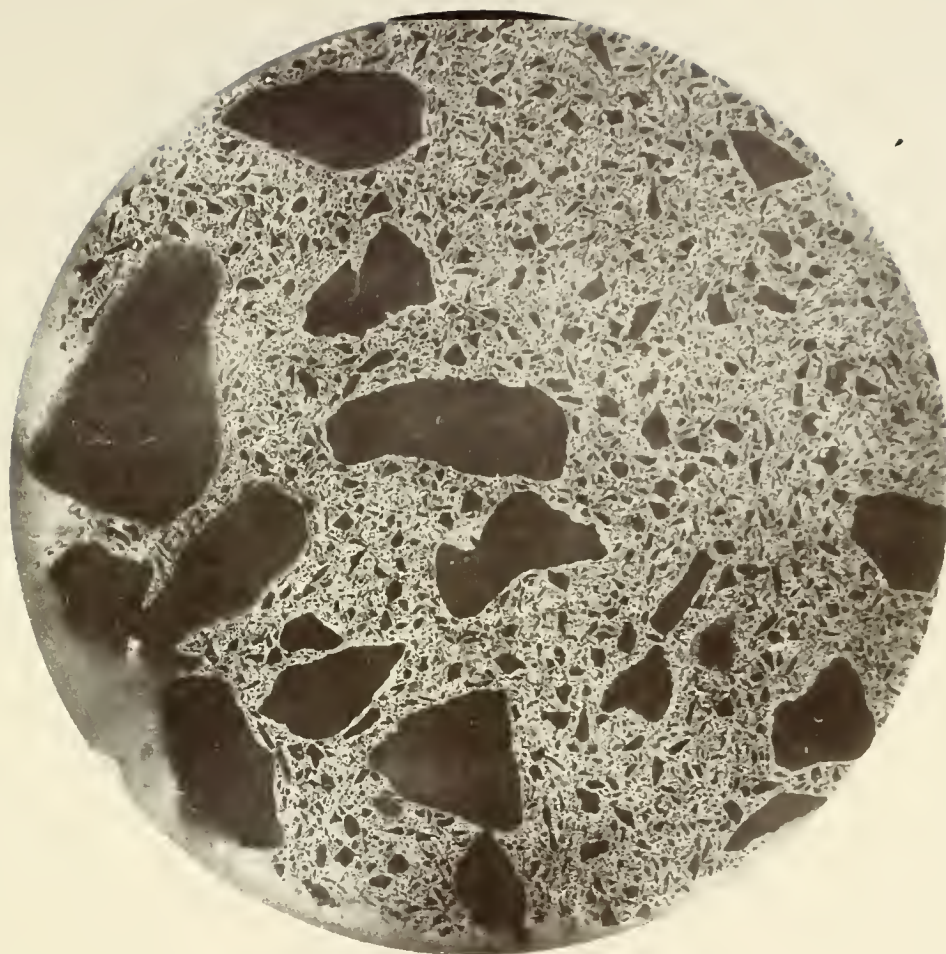


Figure 14 Autoradiograph of Specimen With 40 Percent
Coarse Aggregate and 60 Percent Fine Aggregate



Figure 15 Autoradiograph of Specimen With 30 Percent
Coarse Aggregate and 70 Percent Fine Aggregate

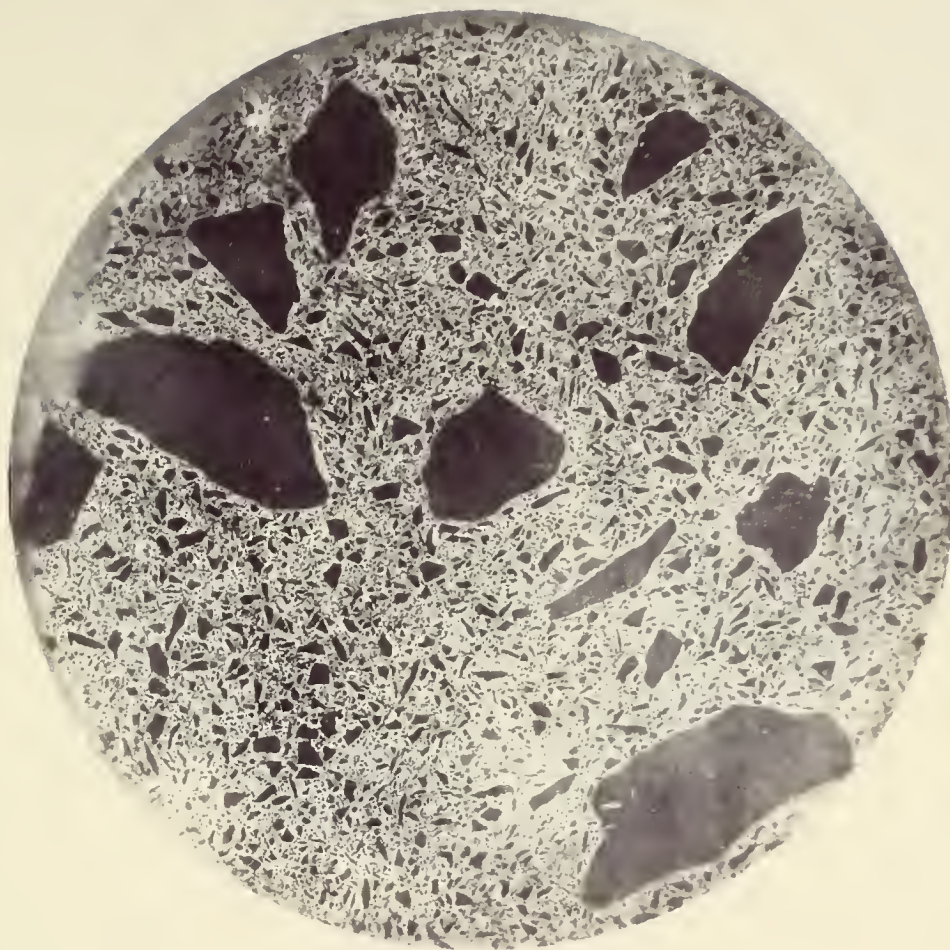


Figure 16 Autoradiograph of Specimen With 20 Percent
Coarse Aggregate and 80 Percent Fine Aggregate

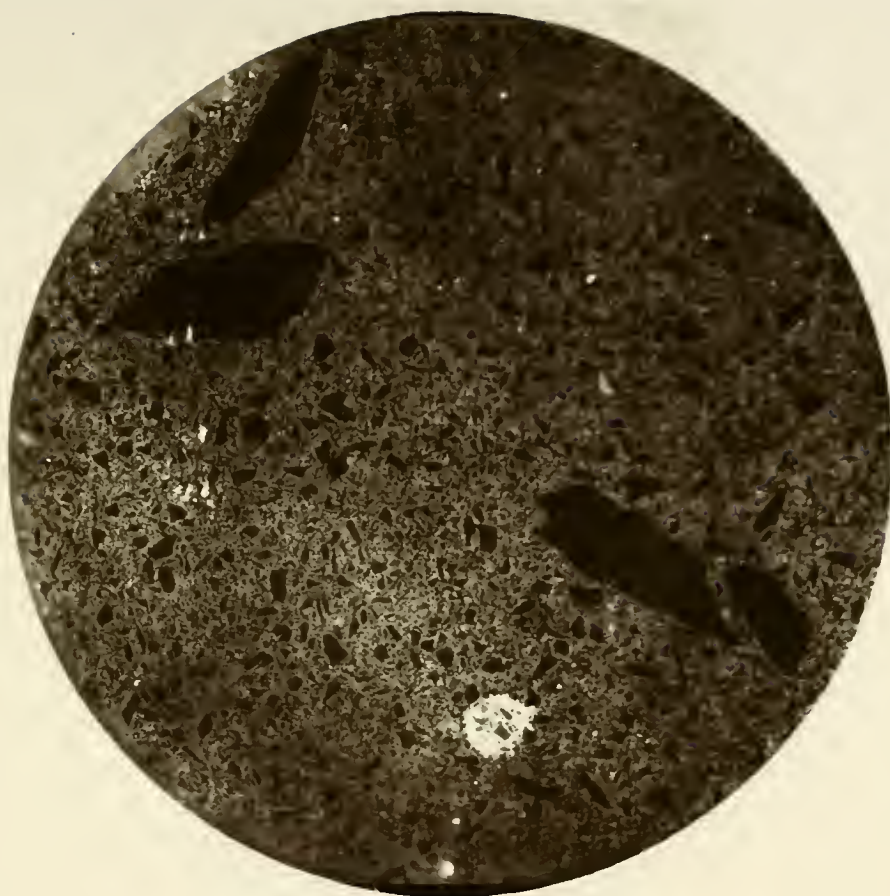


Figure 17 Autoradiograph of Specimen With 10 Percent
Coarse Aggregate and 90 Percent Fine Aggregate

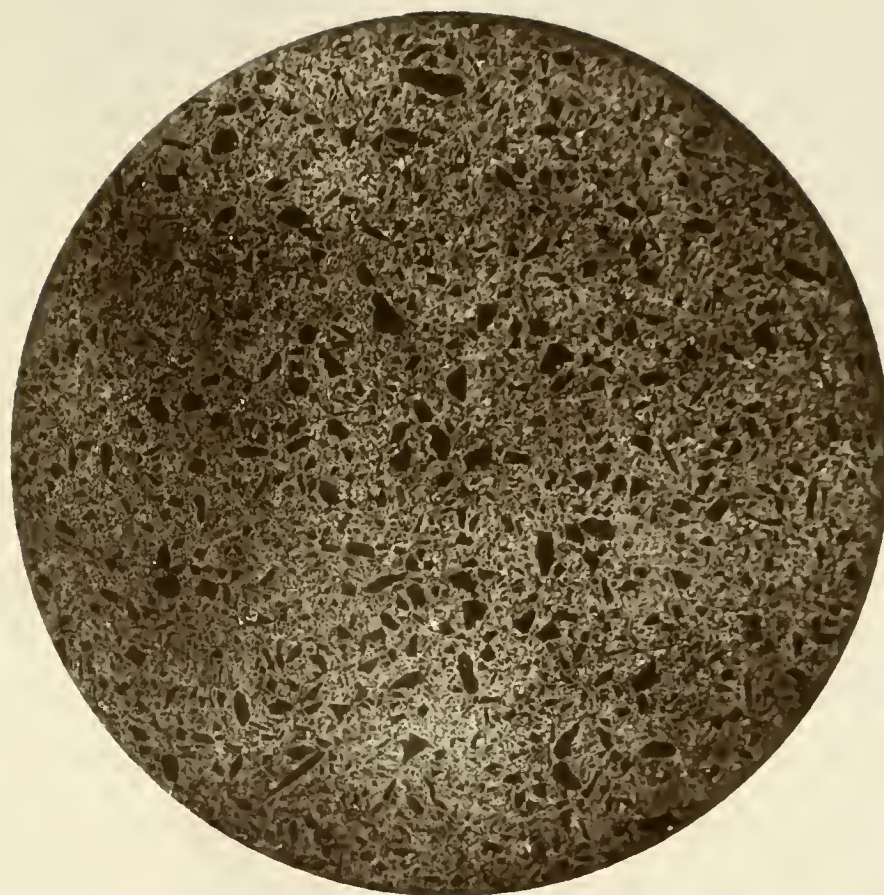


Figure 18 Autoradiograph of Specimen Containing All
Fine Aggregate

using 80 grams of filler and 60 grams of asphalt. This specimen was encased in a four-inch plaster of paris cylinder, cut and polished. The autoradiograph of this specimen is shown in Figure 19. The texture of the autoradiograph indicates even distribution of the filler material throughout the asphalt.

A series of specimens was made using the following aggregate weights; fraction between the 1/2 and 3/8 inch sieves, 460 grams; fraction between the No. 16 and No. 30 sieves, 460 grams; filler fraction (as above) 80 grams; and asphalt portion (as above) 60 grams. This made the asphalt content 6 percent by weight of the aggregate. The first specimens were made with the Wyoming urananite as filler and with limestone for the other two fractions. The second group was made with the No. 16 to No. 30 size fraction of Wyoming urananite and the other two fractions of the limestone. Due to the fact that the emphasis here was on aggregate distribution in the smaller sizes no samples were made using the urananite for the coarse fraction. The autoradiograph of a specimen where the filler was the urananite and the balance of the aggregate was limestone is shown in Figure 20. Figure 21 shows the autoradiograph for a specimen in which the No. 16 to No. 30 size was radioactive. The points of excessive lightness are the places where the alpha particles impinged upon the film.

A group of specimens was made to see if the autoradiographic technique could be employed to study changes in asphalt-aggregate distribution that occur when mixing time and viscosity of the asphalt are varied. For this part of the research a modified Hobart mixer was used for all the mixing as opposed to the hand mixing that was used for all the preceding mixes.

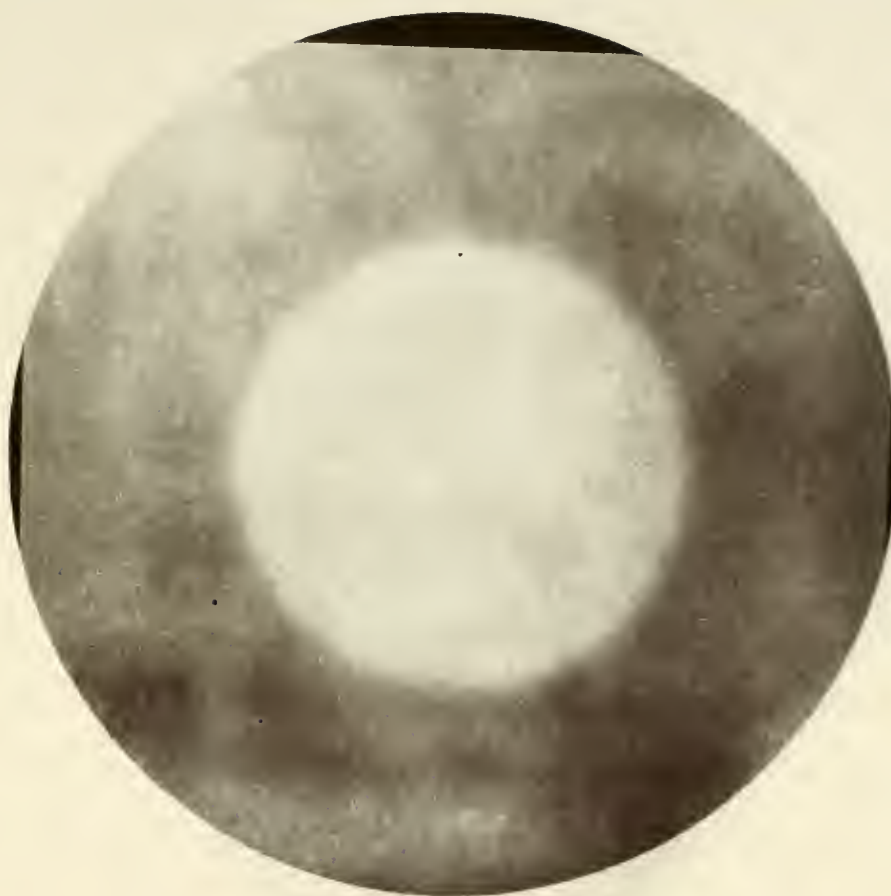


Figure 19 Autoradiograph of Asphalt and Wyoming
Urananite Filler

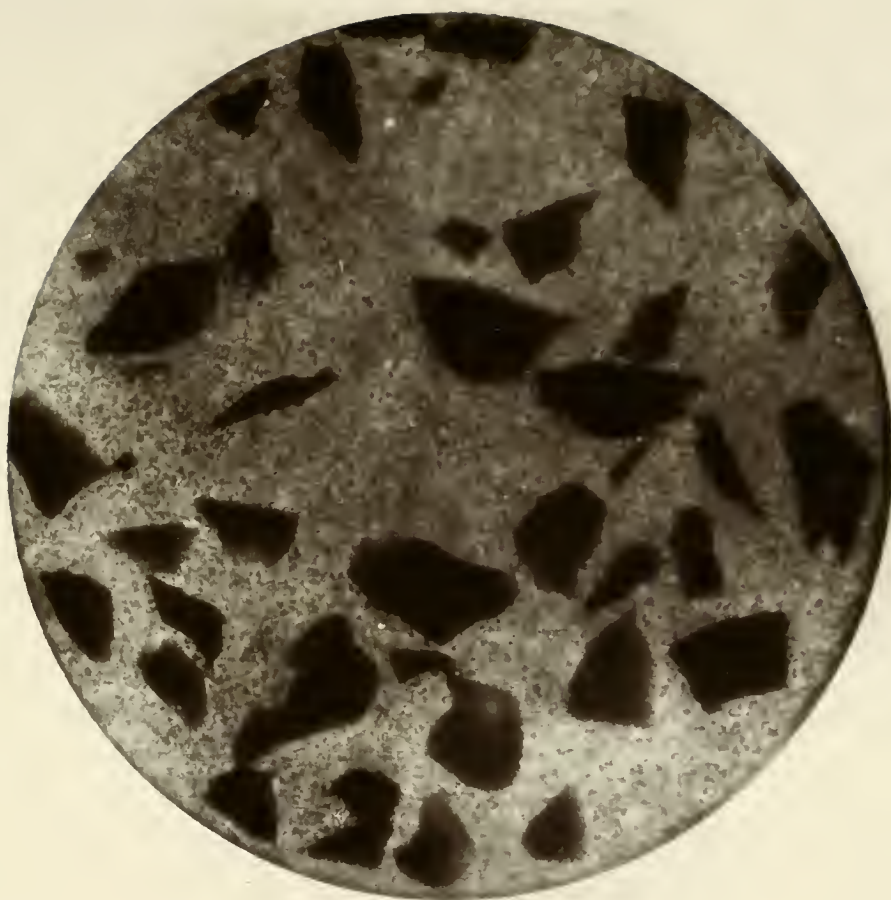


Figure 20 Autoradiograph of Specimen Containing Wyoming
Uraninite Filler

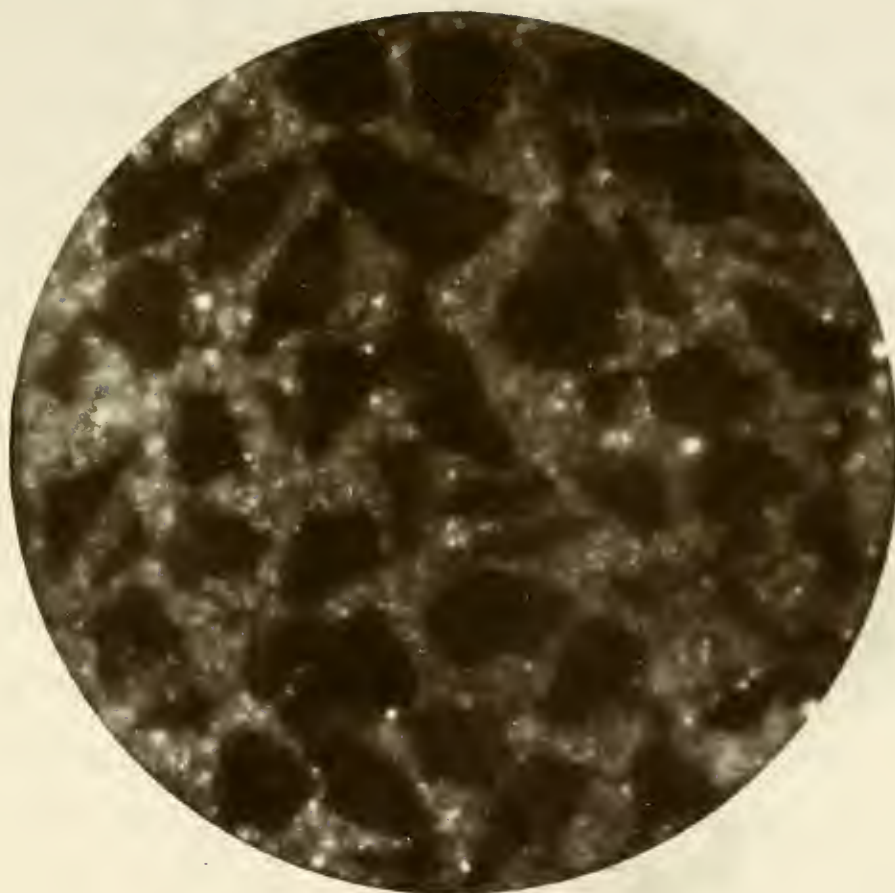


Figure 21 Autoradiograph of Specimen Containing Wyoming Urananite Between the No. 16 and No. 30 Sieves

From the coarse aggregate-fine aggregate mixtures that were used in the portion of this study involving the effect of aggregate gradation, two combinations were chosen for this mixing-time, asphalt viscosity phase, the 80 percent coarse aggregate and the 20 percent fine aggregate mix for one, and the 30 percent coarse aggregate and 70 percent fine aggregate for the other. In the autoradiographs for this portion of the work, the crosshatching in the 80-20 mix as before, shows the hardened Portland cement slurry used to help in the cutting and polishing phases of specimen preparation.

Mixing times of 15, 30, 60, and 90 seconds were used. The aggregate appeared to be completely coated at the end of the 60 seconds of mixing. However, a 90 second mixing time was used to insure complete coverage. The autoradiographs of this series of tests are shown in Figures 22 through 29. The mixing temperature for this portion of the research was 275°F.

A comparison was made between mixes made by the Hobart mixer and those made by hand. The coarse mixture (80-20) was used and hand mixed until all the pieces were coated. The autoradiograph for the specimen made from the hand-mixed sample is shown in Figure 30 for comparison.

Another group of specimens was made using the same aggregate distribution as the above (80-20 and 30-70) and the 90 second mixing time. The variation here was asphalt viscosity produced by varying the mixing temperature. The temperatures used were 225, 250 and 275°F. An attempt was made to use 200°F, but the asphalt was too viscous to mix with the aggregate. The autoradiographs of this series are shown in Figures 31 through 36.

DISCUSSION OF RESULTS

A considerable amount of effort went into the developing of a technique for obtaining good definition in the autoradiographs. The first reaction when viewing the autoradiograph is that normal photography will show as much

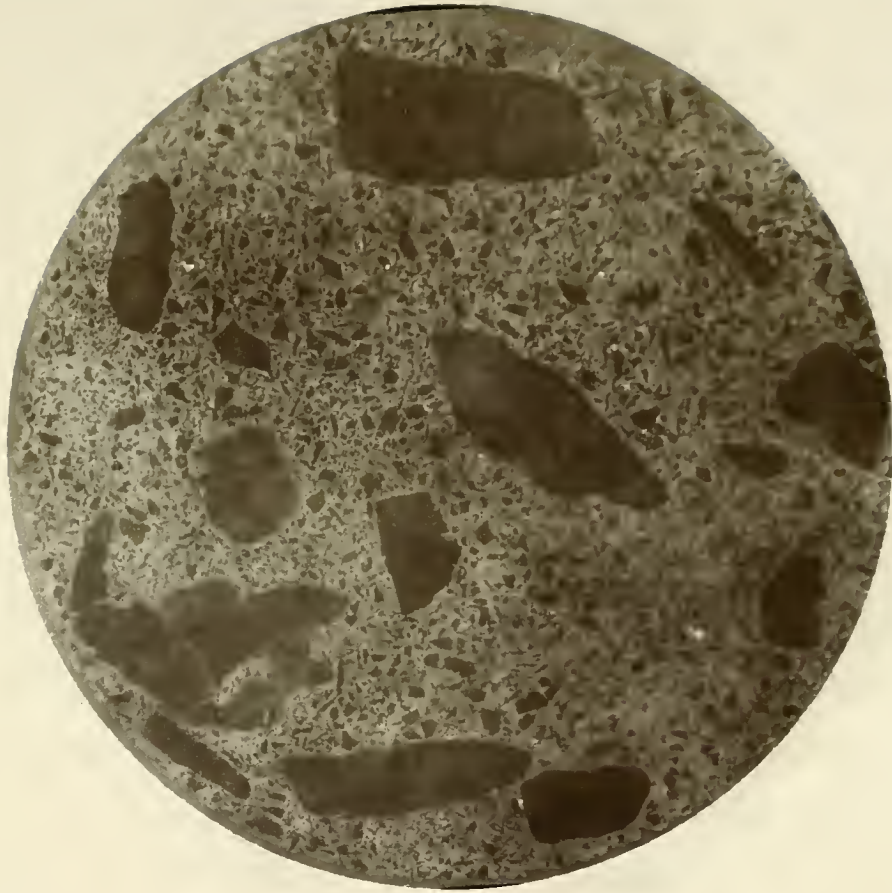


Figure 22 Autoradiograph of the 30 Percent Coarse
Aggregate — Sample Mixed for 15 Seconds

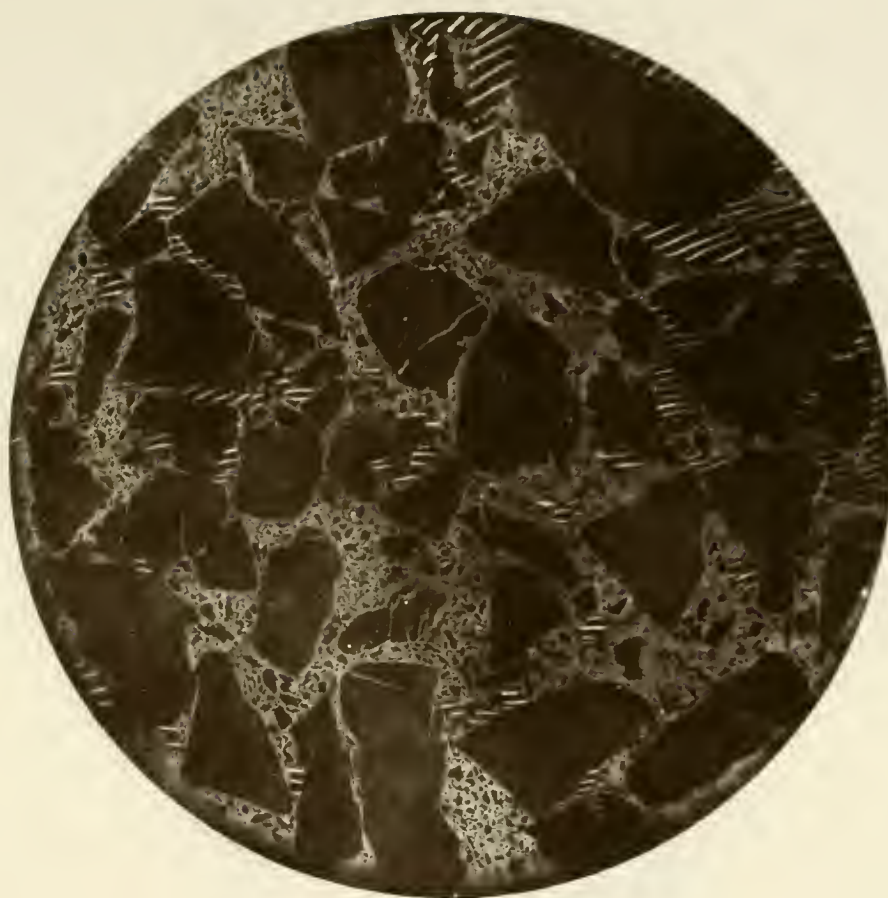


Figure 23 Autoradiograph of the 80 Percent Coarse
Aggregate — Sample Mixed for 15 Seconds



Figure 24 Autoradiograph of 30 Percent Coarse Aggregate
Sample Mixed for 30 Seconds



Figure 25 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 30 Seconds

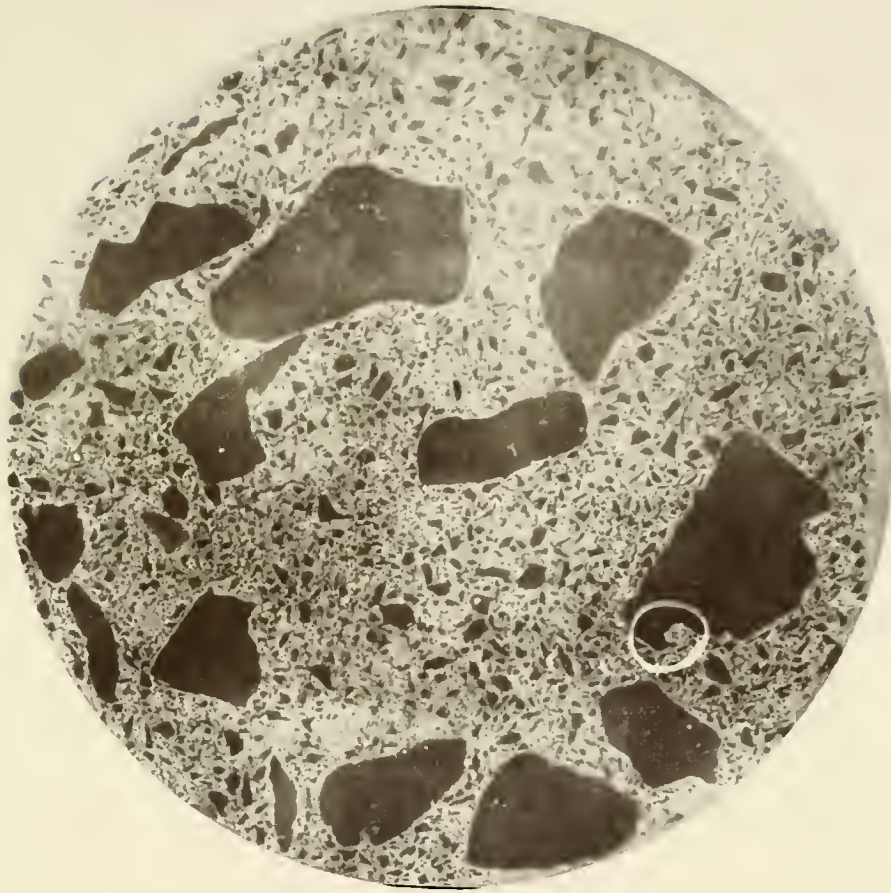


Figure 26 Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed for 60 Seconds

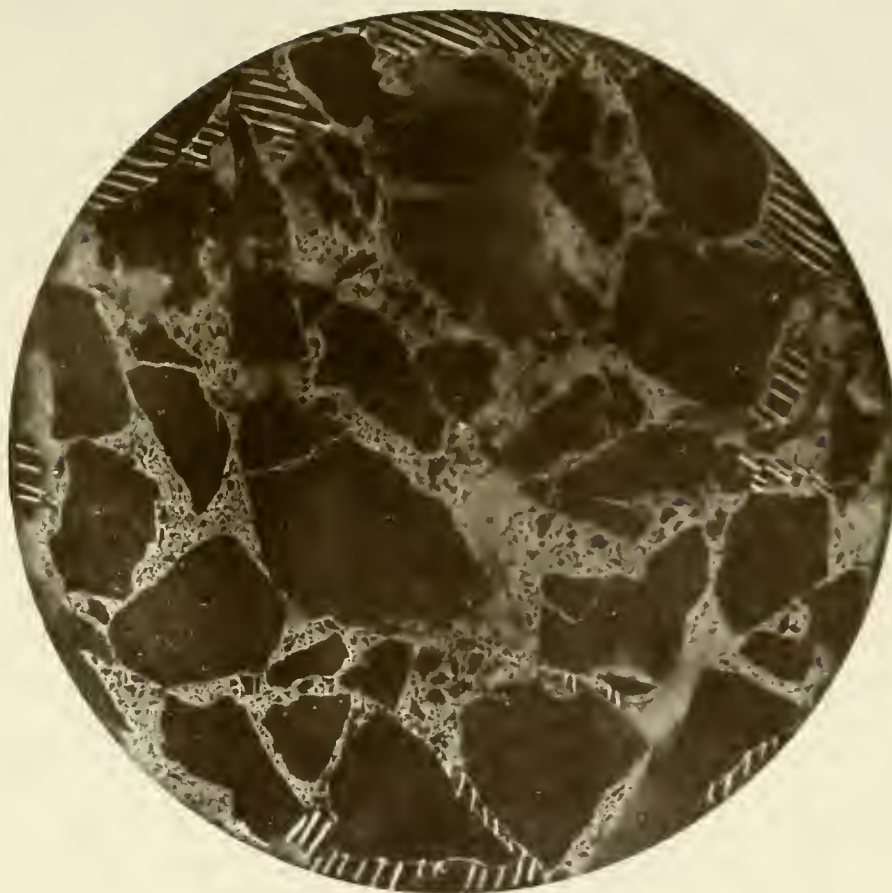


Figure 27 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 60 Seconds

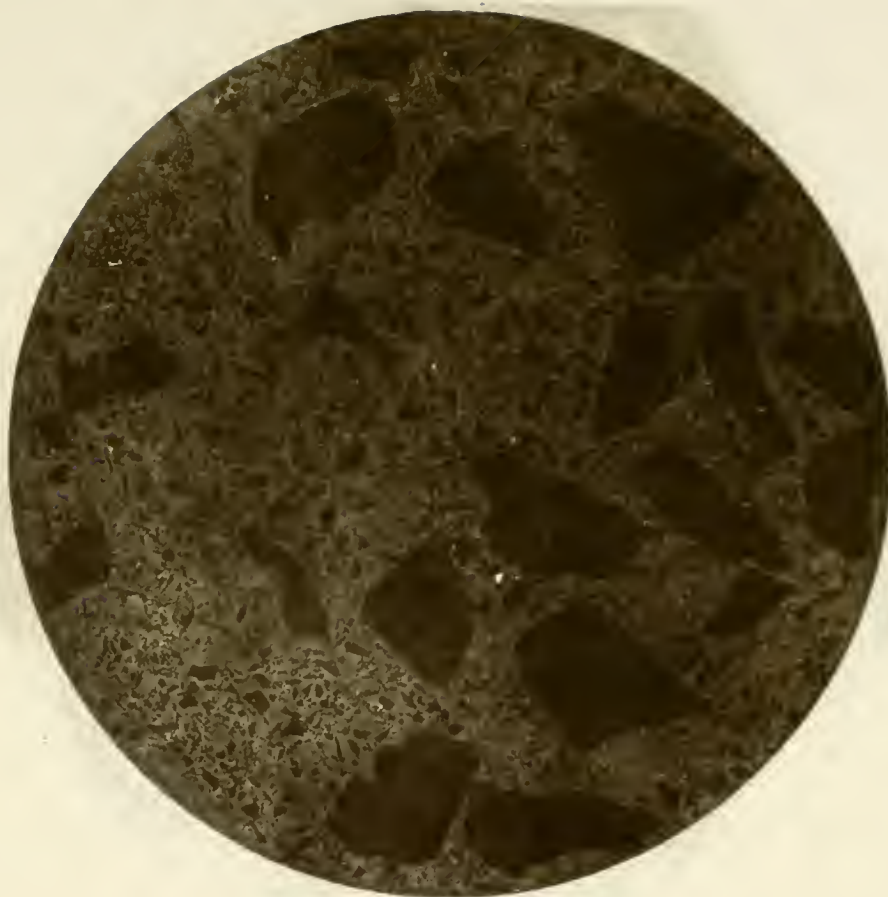


Figure 28 Autoradiograph of 30 Percent Coarse Aggregate —
Sample Mixed for 90 Seconds

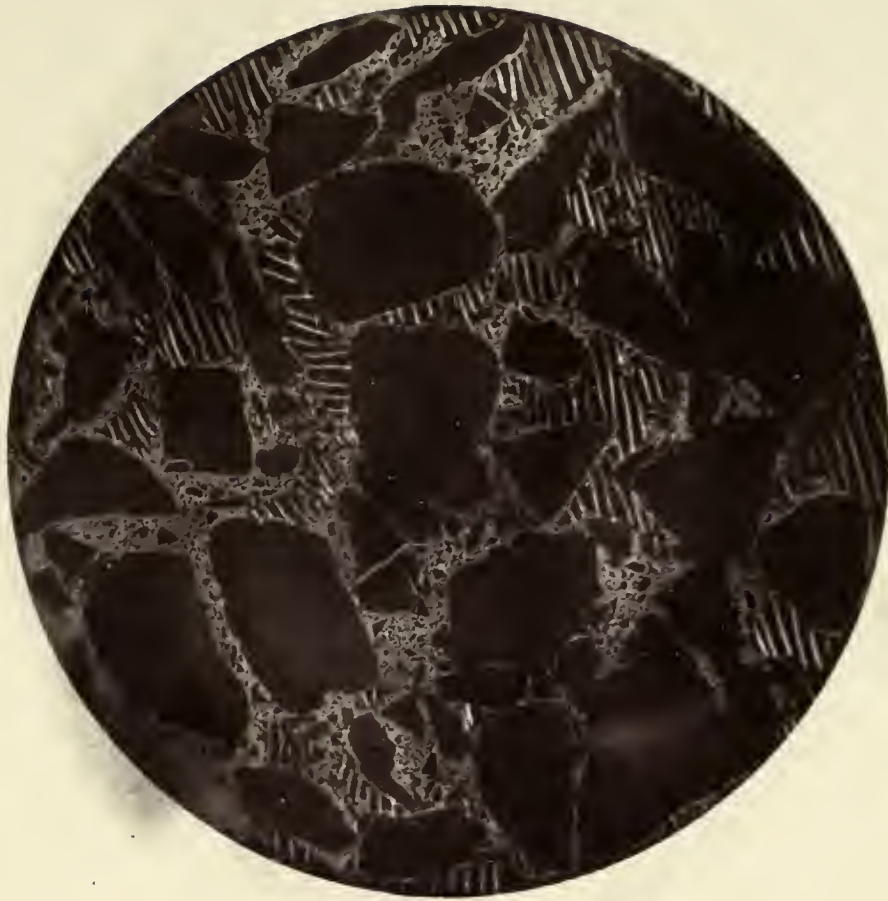


Figure 29 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 90 Seconds



Figure 30 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed by Hand

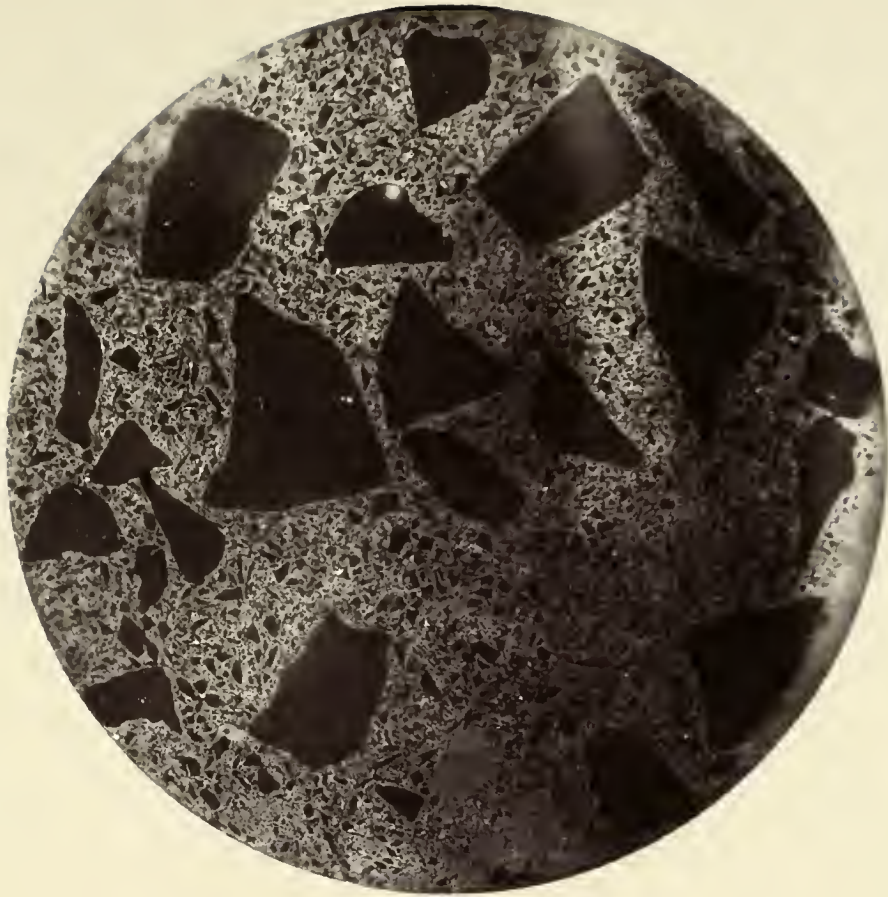


Figure 31 Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed at 225°F for 90 Seconds

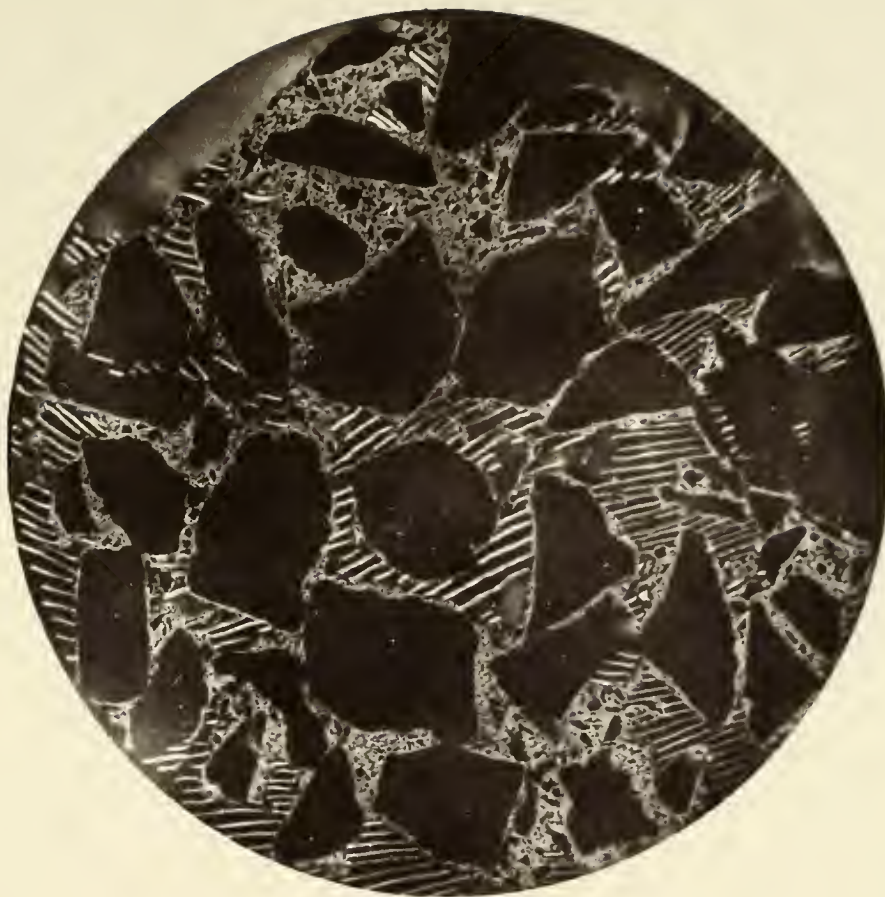


Figure 32 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed at 225°F for 90 Seconds

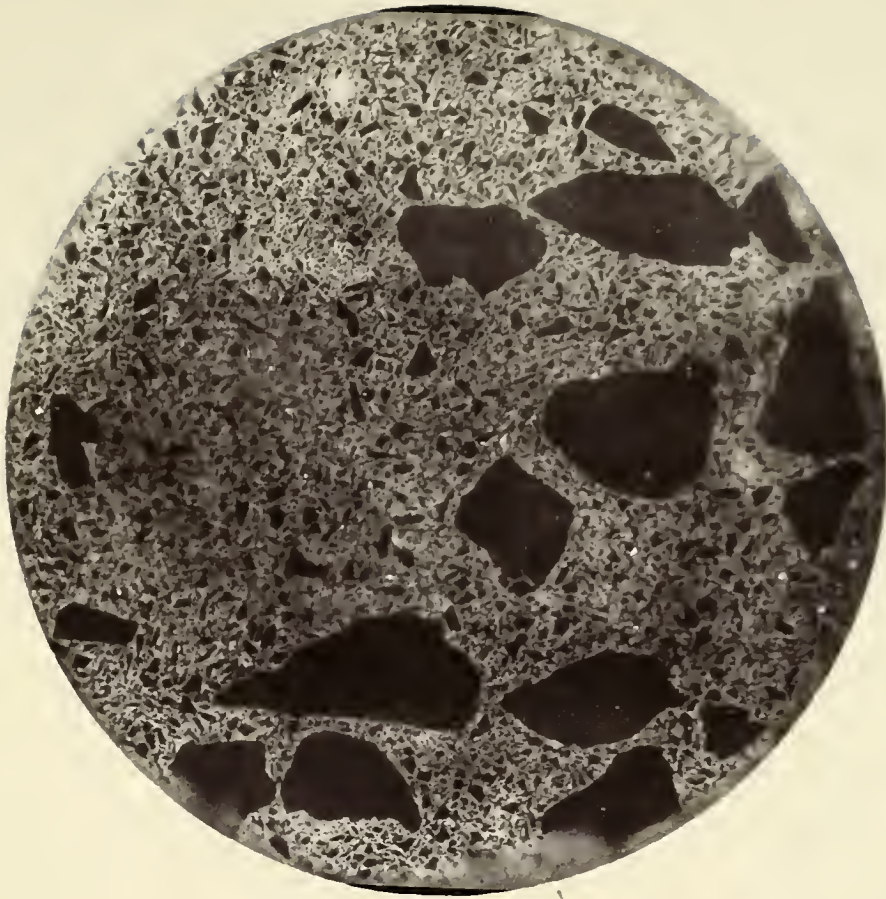


Figure 33 Autoradiograph of 30 Percent Coarse Aggregate —
Sample Mixed at 250°F for 90 Seconds



Figure 34 Autoradiograph of 80 Percent Coarse Aggregate —
Sample Mixed at 250°F for 90 Seconds

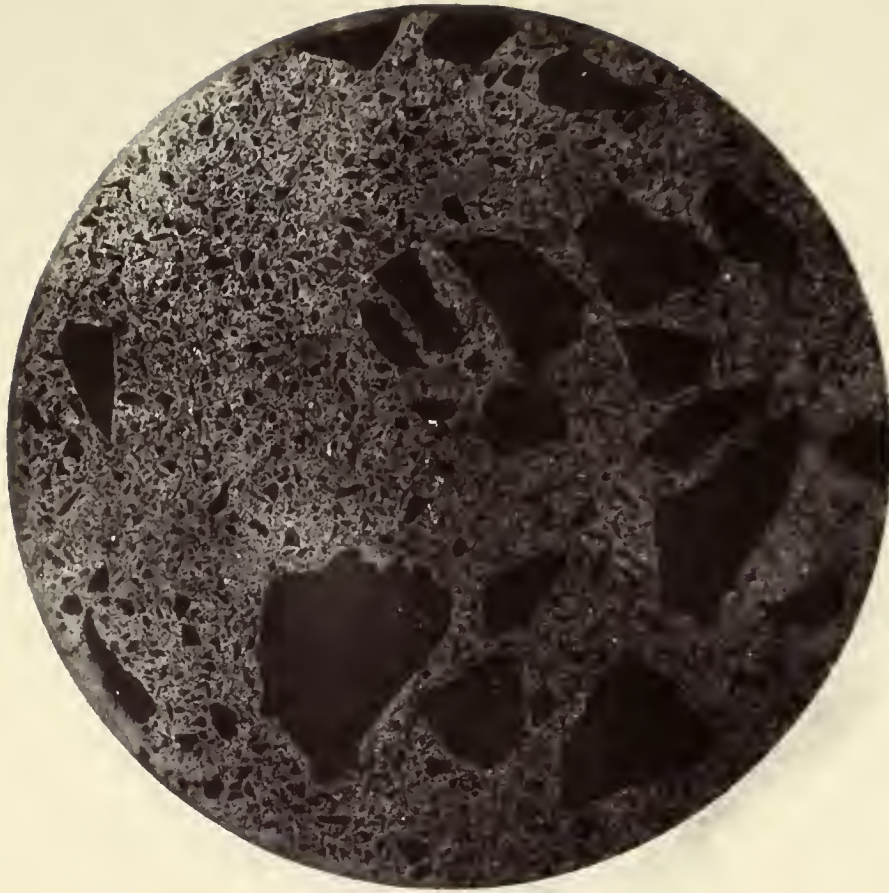


Figure 35 Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed at 275°F for 90 Seconds

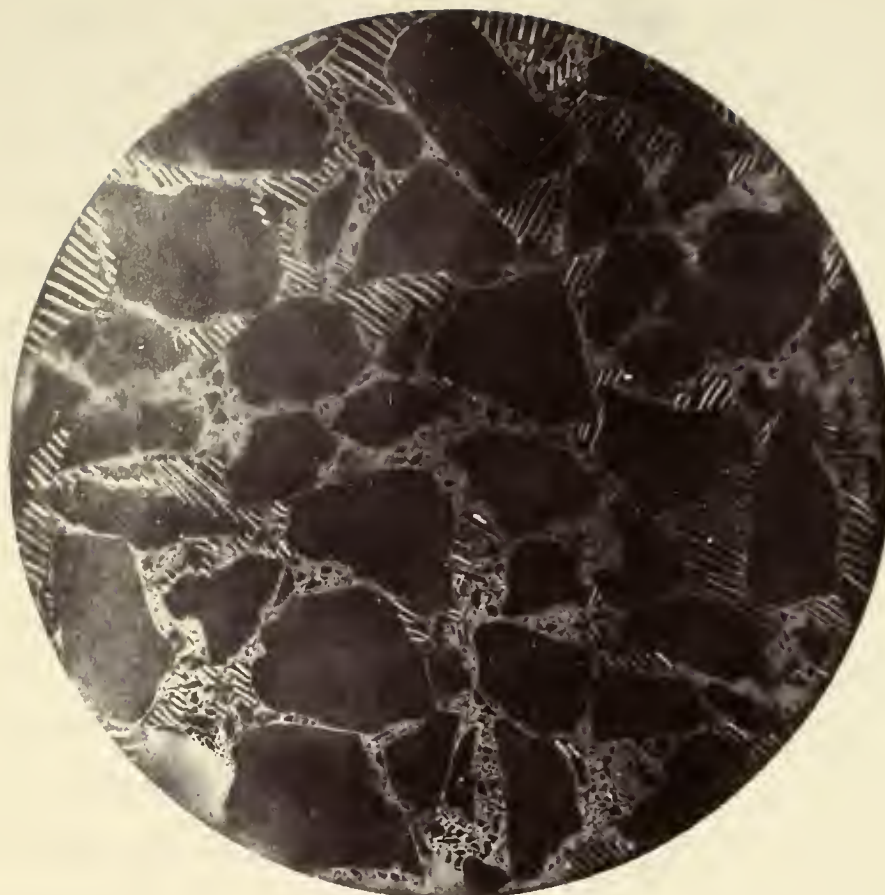


Figure 36 Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed at 275°F for 90 Seconds

detail as the autoradiograph. However, closer examination of the two shows that broken aggregate is visible in the autoradiographs only when radioactive asphalt migrates into the break. The natural photographs show all aggregate pieces that were broken or scarred during the compaction and cutting processes. The crevices due to cutting were so deep that the polishing did not remove the irregularities. This comparison can readily be seen in Figure 4 (the autoradiograph), and Figure 5 (the regular photograph). Both pictures are made from the same specimen face. Thus the autoradiographic technique offers a means by which fracture due to mixing and compacting, and a fracture due to aggregate breakage under load, or from cutting or some other treatment, might be distinguished.

A better comparison can be made where other than a light-colored aggregate is used. When a dark aggregate is used, such as a trap rock, the definition of the autoradiograph does not change, whereas the normal photography will lose definition until asphalt and aggregate are almost indistinguishable. This comparison can be seen in the two companion pictures (Figure 6 and Figure 7) when the Belgium black marble was used as the aggregate. In the autoradiograph (Figure 6) the broken aggregate with the asphalt forced into the crack is plainly visible, but in the photograph (Figure 7), this cannot be seen with much clarity. Of course there are all shades of comparison between these two and probably there are situations where the color and surface texture of the asphalt and aggregate very closely approach each other and make differentiation by ordinary photography even more difficult.

The next comparison made is that between the photomicrograph of the sample face and the photomicrograph of the autoradiograph. In Figure 2 the photomicrograph of the sample face is presented and in Figure 3 the photomicrograph of the autoradiograph is shown. The grain of the original auto-

radiograph is visible in the photomicrograph, but even with this deterrent better resolution is shown than in the regular photomicrograph.

The larger pieces of aggregate are about equally distinguishable in Figures 2 and 3. However, as the size of the aggregate decreases the definition is lost, first in Figure 2. The very small aggregate particles, the minus No. 100 and No. 200, can be measured in Figure 3.

Although the data are not presented herein, one of the important phases of this research was to study the effect of different types of film emulsions. The X-ray film did not show as much definition as the NTB film does, but the sensitivity of the X-ray film was greater than the NTB film. The larger the emulsion grain size the greater the sensitivity and the poorer the definition. The X-ray emulsion has a fairly large grain size as compared to the NTB emulsion. However, the X-ray film has a grain size that works well as far as sensitivity is concerned and yet has good definition. The other films used were not as adaptable as the two discussed above although the Du Pont emulsion could be used in sheet film.

Considering the fact that it was the least expensive of all films used, the X-ray film proved to be the best all around autoradiographic film. If in some particular cases an added amount of definition was needed NTB sheet film could be used as a supplement. The X-ray film was used for the volume work until such time as specific high resolution was needed to show or give emphasis to a particular spot on a specimen. Then the use of the NTB film was warranted. In addition to costing about 45 times as much, the NTB film takes at least 10 times the exposure time as does the X-ray film.

The effect of mineral type of the asphalt-aggregate interface was also shown by use of the autoradiograph. In Figure 15 the largest piece of aggregate on the specimen face is a piece of limestone and chert. Under



Figure 37 Photograph of a Sample with a Piece of Aggregate
That is Part Limestone and Part Chert

magnification it was seen that there are discontinuities along the asphalt-chert interface. Figure 37 is a photograph of this same specimen face showing the same aggregate piece as well as the different colors of the two mineral types. However, magnification showed that this photograph does not contain the detail shown in Figure 15. Thus the autoradiographs should be useful in studying the interface between various asphalts and different mineral types.

The asphalt-aggregate distribution studies considered the effect of aggregate gradation, use of radioactive aggregates, and effect of asphalt viscosity and mixing time variables. These all seem to be closely inter-related as far as this research is concerned.

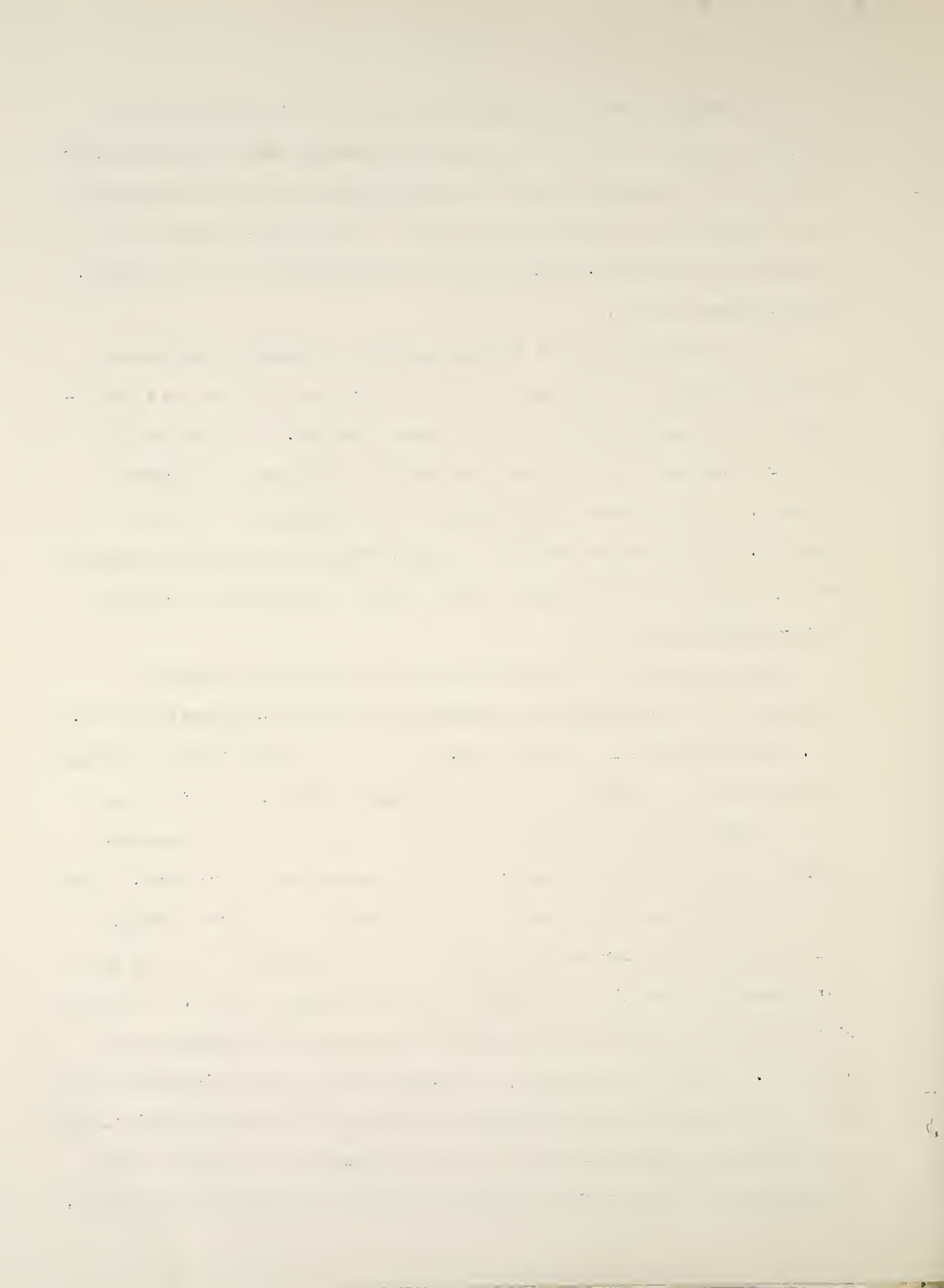
In the aggregate gradation phase of the research two aggregate fractions were mixed with the percentage of asphalt that gave maximum stability in the Marshall test (fine aggregate), or the amount of asphalt the aggregate could hold at the particular mixing temperature (coarse aggregate). This gave about the optimum asphalt content for each fraction. If the asphalt had any preference for one particular fraction in the mixture it would concentrate around this fraction and be noticed in the autoradiographs. However this preference for a particular fraction was not observed.

In Figures 8 through 18 a series of mixes is shown beginning with 100 percent coarse aggregate. (The apparent fines in Figure 8 are due to aggregate degradation during mixing and compacting.) As the fines are added to the mix there does not seem to be any change of the film thickness of the asphalt around the coarse pieces. In Figure 8 the outline of each piece by the light line shows this film thickness. In each of the following figures (Figures 9 through 19) as the percentage of fines increases the fines mix with the asphalt as a mortar and then coat the coarse pieces. By the time 40 percent of the total mix is fine aggregate, the coarse fraction is dispersed in the mortar of the fine aggregate and the asphaltic cement.

The coating on the coarse pieces is not a film of asphaltic cement alone, but a matrix of the mortar consisting of asphalt with the fine fractions embedded in it. In Figure 17 this is quite pronounced due to this mix having only 10 percent of the coarse fraction in it. In this figure only four coarse pieces can be seen, each embedded in the matrix of the fine aggregate-asphaltic cement mortar.

It is also noted that as the fine aggregate is added to the coarse fraction the fines always adhere to the coarse fraction or appear in a concentration in the interstitial part of the coarse fraction. This adherence to the coarse fraction is part of the so-called film thickness of the coarse aggregate. It is composed of the asphalt and the finer parts of the fine aggregate. When there are some of the coarser fractions of the fine aggregate present, they tend to form a mortar and fill the interstitial part of the coarse-aggregate mix.

The specimens which contained radioactive aggregate as only one fraction of the aggregate gradation showed an even asphalt-aggregate distribution. When the material passing the No. 200 sieve was radioactive it combined with the asphalt in much the same way as did the sulfur-35. If this mortar had any preference for an aggregate fraction it would show in an autoradiograph as a light area at the surface of the aggregate size it preferred. This did not occur with either the radioactive aggregate or the tagged asphalt. Therefore when the filler material combines with the asphalt, the mortar thus formed acts very much like the asphalt itself when placed in a mix. In Figure 20 the minus 200 material was radioactive and the other two fractions were limestone. In this autoradiograph, the light spots are due to alpha particles and are not to be confused with a high concentration of the beta particles. The uniformity of the autoradiograph shows no concentration, but does show even distribution of the radioactive material throughout the specimen cross section.



In Figure 21, the No. 16 to No. 30 fraction was the radioactive aggregate and the other two fractions were limestone. Again there is an even distribution of the radioactivity. Some of the coarse aggregate pieces, shown in Figures 20 and 21, do not have a sharp line of demarcation because the energy level of the beta particles from the aggregate is higher than that from the sulfur-35. An even distribution of radioactive aggregate is shown in the three autoradiographs (Figures 19, 20, and 21) made from specimens that were made with one fraction of the radioactive aggregate.

Mixing time and asphalt viscosity were varied to see if the autoradiographic technique would reveal differences in asphalt-aggregate distribution resulting from these variations. In Figures 22 and 23 a 15-second mixing time was used. After this mixing period most of the coarse pieces still were not completely coated. However, after they were compacted there was evidence that almost complete coating was accomplished. In Figure 22 the 30 percent coarse aggregate specimen shows that only at points of coarse aggregate contact does a coating seem to be lacking. In Figure 23 wherever the coarse aggregate was not coated, but was in contact with some other coated piece, or matrix, a coating exists. If no contact was made the aggregate remained uncoated. (See large piece at right hand side of autoradiograph, where there is no change between the aggregate piece and the Portland cement slurry). In all the rest of the 30 percent coarse aggregate autoradiographs, the coarse aggregate is coated (see Figures 24, 26 and 28).

In Figure 25 (mixing time 30 seconds) some contact points between the coarse particles are not coated but generally speaking all the pieces are coated. In Figure 27 (60 seconds mixing time) all the pieces are coated, and most of the contact points are coated. The only place where coating does not occur is where aggregate degradation occurred. These are filled if there was



some of the matrix close enough to them for the hydraulic action of the compaction process to force the asphalt into the fracture. In Figure 29 all pieces are coated except a few contact points. The contact points are not coated because the high pressures at these points of contact forced the asphalt away during the compacting process. In Figure 30 even more coverage is shown than in the machine-mixed samples, but in this case the sample was mixed by hand until all the pieces were coated. Notice **also** that in the hand-mixed sample more of the coverage is of the matrix type.

In Figure 26 the circled part of the autoradiograph is a hook on a large piece of the coarse aggregate. Forces caused by the compactive effort of the Marshall hammer have forced the matrix into this hook showing the extent of the hydraulic action produced by compaction. This coupled with the above information indicates that in a dense-graded mix, even though there is not complete coverage at the end of the mixing operation, coverage is obtained when the material is compacted.

The asphalt viscosity variations were achieved by varying mixing temperature. In Figure 31 (225°F) there are some balls of fines and asphalt that were not broken down during the mixing operation. When the temperature was increased to 250°F only one very small ball of this material could be found (Figure 33). When the temperature reached 275°F none of this material was left unmixed.

Autoradiographs of mixes that incorporated a change of viscosity as the variable are shown in Figures 32, 34 and 36. These autoradiographs show that as the asphalt became more viscous a thicker film of matrix covered the coarse aggregate pieces.

CONCLUSIONS

The results obtained from the analysis of the test data appear to justify the following conclusions. However, it should be realized that in general the conclusions are applicable to the particular asphaltic mixtures and sample preparations that were used in this research.

The autoradiographic technique is very adaptable to the study of the asphalt-aggregate distribution in an asphaltic mixture. Good definition can be obtained using X-ray film emulsions, but better definition can be achieved by the use of the NTB autoradiographic stripping film emulsions. The use of a weak beta emitter gives better definition than the use of a highly energetic beta emitter.

Autoradiographs can be made using many different types of film emulsions. The data collected during the research phase of this investigation proved that the X-ray film worked very well for the multitude of autoradiographs made because of its high sensitivity and its good definition. The NTB film gave better definition, but was less sensitive and more expensive, and should be used only if the extra definition is needed.

Autoradiographs give more detail than regular photographs where the aggregate is dark and it is difficult to distinguish between the color of the asphalt and the aggregate. The autoradiograph will not show the imperfections of the aggregate particles that are caused by the cutting of the sample. Polishing of the sample face will remove some of these imperfections as well as any asphalt that has been deposited on the aggregate during the cutting process, and the aggregate will remain clear in the autoradiograph. Imperfections that occur during the mixing and compaction operation will show when the asphalt has intruded into the crack. Thus the autoradiograph indicates the asphalt-aggregate distribution more precisely than the photograph especially when the aggregate-asphalt colors are very similar.

Film thickness can be observed in open bituminous mixtures. However, when the gradation gives a relatively dense mixture specific film thickness cannot be observed. In this dense condition the fines and the asphalt form a matrix and the coarse pieces are dispersed in the matrix. Therefore, in this dense condition the mixture acts very much like Portland cement concrete in its ground mass to phenocryst relationship. The mixture also resembles Portland cement concrete in appearance of the binder-aggregate distribution. In dense-graded mixtures, no films as such are formed on any particular size of the aggregate.

Even though complete coverage might not be achieved at the conclusion of mixing, in a graded mixture any portion of the aggregate not covered tends to be covered during the compaction process. In open mixes this will not occur because at points of high contact pressures between points of the aggregate, the asphalt will be forced away from the aggregate. Thus, in speaking of mixing efficiencies of bituminous mixes it is important to separate open and graded mixes.

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